FEDERAL UNIVERSITY OF ITAJUBÁ MECHANICAL ENGINEERING INSTITUTE

EXPERIMENTAL ANALYSIS OF ECO-FRIENDLY MORTAR WITH INDUSTRIAL WASTE

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EXPERIMENTAL ANALYSIS OF ECO-FRIENDLY MORTAR AND CONCRETE WITH INDUSTRIAL WASTE

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A thesis presented to the Postgraduate Program of Mechanical Engineering, from Mechanical Engineering Institute of the Federal University of Itajubá, as a requirement to obtain the title of Master in Mechanical Engineering

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I dedicate this work to my mother and father who have always been by my side at all times.

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"No one will hit as hard as life. However, it's not about how hard you can hit, it's about how hard you can be hit and keep moving forward. That's how victory is won."— Rocky Balboa

Abstract

The use of industrial waste in cementitious materials is a major challenge for the construction industry. In many sectors, the waste usually presents heterogeneity in terms of volume and production composition, which hinders its use on a large scale. However, cementitious materials such as laying and covering mortars, as well as concrete, if properly proportioned, can tolerate a certain amount of waste. To ensure a systematic and accurate analysis of the system formed by waste plus cementitious composites, it is necessary to use semi-empirical methods that combine experimental results with mathematical models. The present study presents an analysis of mortars with polyurethane (PU) industrial waste and foundry exhaust sand (FES). This analysis is presented in different scenarios. First, a bibliometric survey was created to map the main authors, journals and keywords used. Second a study of the mechanical, physical behavior and workability of conventional mortars with partial replacement of natural sand with polyurethane waste, in this model a Design of Experiments (DOE) was used to plan and analyze the responses. Third, a delimitation of the range of percentages used for the control variables of the second experiment was performed, these variables were percentage of sand by cement (S/C), coarse sand content (SC), percentage of substitution of FES by natural sand (FES), percentage of substitution of PU by natural sand (PU). In this way it was possible to perform a response surface (RSM) through a quadratic model by means of the developed DOE. The fourth scenario of the study involved the dynamic analysis of mortars with waste in which it was possible to obtain the dynamic behavior of the mortar structure. Numerical models were created for all the situations tested, these models proved capable of providing data to adjust the predictive indices that influence the mechanical, physical and dynamic quality of the mortars tested. The results serve as a basis for the development of more sophisticated and optimized methods for dosing and incorporation of waste in cementitious composites on a large scale

Keywords: DOE; mortar; RSM; foundry exhaust sand; polyurethane; response surface methodology; artificial neural network.

Resumo

A utilização de resíduos industriais em materiais cimentícios é um grande desafio para a indústria da construção. Em muitos setores, os resíduos geralmente apresentam heterogeneidade em termos de volume e composição de produção, o que dificulta sua utilização em larga escala. No entanto, materiais cimentícios como argamassas de assentamento e revestimento, bem como concreto, se bem dosados, podem tolerar uma certa quantidade de resíduo. Para garantir uma análise sistemática e precisa do sistema formado por resíduos mais compósitos cimentícios, é necessário o uso de métodos semiempíricos que combinem resultados experimentais com modelos matemáticos. O presente estudo apresenta uma análise de argamassas com resíduos industriais de poliuretano (PU) e areia de exaustão de fundição (FES). Esta análise é apresentada em diferentes cenários. Primeiramente, foi criada uma pesquisa bibliométrica para mapear os principais autores, periódicos e palavras-chave utilizadas. Segundo um estudo do comportamento mecânico, físico e trabalhabilidade de argamassas convencionais com substituição parcial de areia natural por resíduos de poliuretano, neste modelo foi utilizado um Projeto de Experimentos (DOE) para planejar e analisar as respostas. Terceiro, foi realizada uma delimitação da faixa de porcentagens utilizadas para as variáveis de controle do segundo experimento, essas variáveis foram porcentagem de areia por cimento (S/C), teor de areia grossa (SC), porcentagem de substituição de FES por natural areia (FES), percentual de substituição de PU por areia natural (PU). Desta forma foi possível realizar uma superfície de resposta (RSM) através de um modelo quadrático por meio do DOE desenvolvido. O quarto cenário do estudo envolveu a análise dinâmica das argamassas com resíduos em que foi possível obter o comportamento dinâmico da estrutura da argamassa. Foram criados modelos numéricos para todas as situações testadas, estes modelos mostraram-se capazes de fornecer dados para ajustar os índices preditivos que influenciam a qualidade mecânica, física e dinâmica das argamassas testadas. Os resultados servem de base para o desenvolvimento de métodos mais sofisticados e otimizados de dosagem e incorporação de resíduos em compósitos cimentícios em larga escala.

Palavras-chave: DOE; argamassa; RSM; areia de exaustão de fundição; poliuretano; metodologia de superfície de resposta; rede neural artificial.

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List of acronyms and symbols

a_i	Describe the vibration amplitude
ANN	Artificial Neural Network
ANOVA	Analysis of variance
BBD	Box-Behnken Design
b_i	Describe the vibration amplitude
CC	Conventional concrete
CFC	Chlorofluorocarbon
CS	Coarse sand content
CT	Curing time
DAQ	Data Acquisition board
DOE	Design of experiments
E	Modulus of elasticity
e	Void index
EDS	Dispersive energy spectroscopy
EMA	experimental modal analysis
f_{ck}	Compressive strength
FES	Foundry exhaust sand
FFD	full factorial design
FFT	Fast Fourier transform
FRF	Frequency response function
FS	Foundry sand
FTIR	Fourier transform infrared spectroscopy
GFFD	General full factorial design
IPS	Interparticle Separation
ITZ	Interfacial Transition Zone
k	Number of factors
MF	Modulus of fineness
MPUFS	Mortar with polyurethane powder waste and foundry exhaust sand
MSE	Mean Squared Error
MTP	Maximum Paste Thickness
n	Number of replicas
NS	Natural sand
PU	Polyurethane powder waste content
PW	Plastic waste
R	Model response
R^2	Coefficient of determination
RSM	Response surface methodology
SSC	self-compacting concrete
S/C	Sand and binder ratio
SEM	Scanning Electron Microscopy
SF	Silica fume
Sp	Spreanding
TED	Taguchi Experimental Design
TEI	Impulse Excitation Technique
U_{max}	potential energy at maximum displacement

VSA	Volumetric Surface Area
w/c	Water by cement
w_a	Water absorption
x_n	Design variable
y_n	Consistency
ΔE	lost energy
ζ	Damping ratio
η	loss factor coefficient
η_r	loss factor
ω_r	resonant frequencies

Contents

1 INTRODUCTION

The world is currently experiencing a new social revolution aimed at increasing awareness about climate issues and the associated dangers that can lead to significant negative changes in our way of life [1]. Many countries are now adopting a new social model that seeks alternatives to traditional industries, aiming to minimize environmental impacts that cause pollution and contamination, thereby mitigating the effects of global warming and the overexploitation of natural resources [2]. Efforts must be directed towards averting the regression of modern civilization by reducing dependence on traditional raw materials. Therefore, the exploration of valid alternatives through recycling and the reclamation of industrial waste appears to be the most promising path forward [3, 4].

This study focuses on two types of waste, PU and FES, which differ in their physical and chemical characteristics but have equivalent negative impacts on the environment [5]. PU, derived from the polymer family, exhibits remarkable versatility and is utilized extensively on an industrial scale due to its various shapes and sizes [6]. This material boasts low production costs, high durability, and finds widespread applications in the construction sector and household appliance industry [4, 7]. Conversely, foundry sand, a by-product of metallurgical processes, known as function sand or green sand, is integrated into molds that undergo multiple reuses during metal part fabrication [8, 9]. During the manufacturing process, sand residues with high surface area become dislodged in the molds and are captured by factory exhaust fans. FES, being airborne residue, is typically finer than conventional exhaust sand [10]. Its disposal poses challenges due to the multitude of possibilities for contaminating local fauna and flora, compounded by its finer texture [11].

The unregulated extraction of natural resources like river sand inflicts irreversible environmental harm. It is imperative that the civil construction industry prioritizes the replacement of traditional natural aggregates. On average, between 25.9 to 29.6 million tons of river sand are extracted annually, leading to biodiversity threats, erosion, altered water flows, floods, reduced freshwater supply, and pollution [12, 13]. The substitution of natural sand with industrial waste can help alleviate the pressure on river sand extraction. Additionally, the unsustainable use of Portland cement-based binders should be partially supplanted by alternative materials possessing pozzolanic properties, such as rice husk, coffee husk, metakaolin, and blast furnace slag residues [14].

Over the past decade, global plastic production has averaged 300 million tons annually, with only 14% of polymer waste being collected for recycling, of which merely 9% is actually recycled and returned to the market [15]. Hence, there is a pressing need for studies aimed at adapting the use of PU waste in civil construction. Numerous ongoing studies explore PU applications in civil construction. For instance, PU is utilized in highway ballasts, resulting in superior freeze-thaw strengths, fatigue resistance, and reduced residual deformation [16]. Briones L. (2020) executed ecological mortars composed of PU and Portland cement, demonstrating their suitability for hospital block and panel construction [4]. Santamaría V. (2020) investigated plaster mortar with PU, yielding composites suitable for residential construction walls [7]. Barnat H. (2018) explored mortars with PU and hydrophobing agents, achieving satisfactory results for various parameters [17].

Foundry sand (FS) exhibits considerable potential for civil engineering applications. Vilenius (2019) investigated the feasibility of incorporating FS into concrete for highway construction, demonstrating its efficacy as a stabilization layer and load-bearing structure [18]. Zhang (2021) conducted independent tests, concluding that FS, when properly mixed and combined with well-graded aggregates, can serve as load-bearing structures [19]. While FS finds widespread application in low-strength materials and as a substitute for fine and coarse aggregate in cementitious composites [20–22], its drawbacks, such as adverse effects on mechanical strength and freeze-thaw resistance in cementitious composite mixtures, require careful evaluation [23].

The optimization of dosage processes in the manufacture of cementitious products like mortars and concrete can be facilitated by statistical approaches [24]. Various successful methods, including artificial neural networks, genetic algorithms, and neuro-fuzzy inference systems, have been employed for optimal modeling in concrete technology [25–29]. Another promising approach is the utilization of Design of Experiment (DOE), a statistical method for planning and analyzing experiment results. DOE reduces the number of experiments, evaluates variable relationships, provides mathematical models, and yields optimal experimental outcomes [30]. Additionally, DOE enables the prediction of results for different parameter sets [31]. Within the DOE framework lies Response Surface Methodology (RSM), widely utilized for industrial procedure optimization [32]. RSM, based on mathematical and statistical principles, not only elucidates variable effects on desired parameters but also illustrates variable interactions via 3D surfaces. Its accuracy in evaluating mechanical, physical, and durability properties of cementitious products is well-established [33].

This research is justified by the urgent need to find sustainable solutions to the challenges faced by the construction industry, especially concerning the management of industrial waste. The growing awareness of the environmental impacts caused by the indiscriminate exploitation of natural resources and the excessive generation of waste is driving the search for innovative alternatives. For instance, replacing natural aggregates with industrial waste, such as PU and FES, not only reduces the demand for finite natural resources but also minimizes the improper deposition of these wastes, thereby mitigating potential environmental harm. Furthermore, the application of semi-empirical methods like Design of Experiments (DOE) and Response Surface Methodology (RSM) provides a systematic and efficient approach to optimizing the dosage and incorporation of these wastes into cementitious composites. This not only ensures the quality and performance of the materials produced but also maximizes the efficiency of industrial processes, reducing costs and waste. Thus, this research not only contributes to environmental sustainability but also promotes technological innovation and the competitiveness of the construction industry.

2.1 Research objective

As presented in the introduction (Chapter 1), cementitious materials such as mortar and concrete based on portland cement, which have industrial waste in their composition, need to have their behavior monitored and accurately evaluated for large-scale use. Thus, the primary objective of this study is to evaluate the mechanical, physical and dynamic behavior of mortars with FES and PU residues through a statistical approach.

Because this objective is broad, it was necessary to develop other studies that were developed during this research. So, some secondary objectives were established to guide the research, these are listed below:

- Develop a review on the topic of cement composite and plastic waste;
- Study the behavior of the mechanical strength and physical properties of Portland cement mortar with polyurethane residue and exhaust foundation sand;
- Provide statistical analysis that proves and measures quantitatively and qualitatively the behavior efficiency of the cementitious composites under study.

2.2 Outline Thesis

Chapter 2 presents a bibliometric review on the subject of cementitious compounds with polymeric residues.

Chapter 3 presents a theoretical and practical study of the mechanical and physical behavior and workability of conventional mortars with partial replacement of natural sand by polyurethane residues. A full factorial DOE was used for planning and analysis of responses.

Chapter 4 presents a theoretical and practical study of the mechanical and physical behavior of conventional mortars with partial replacement of natural sand by polyurethane residues and foundry exhaust sand. A response surface (RSM) was developed to model the responses.

Chapter 5 presents a theoretical and practical analysis of the dynamic behavior of portland cement mortars with partial replacement of natural sand by polyurethane residues and foundation exhaust sand. A response surface DOE was used for response planning and analysis. An artificial intelligence algorithm was also developed to analyze the responses.

2 ANALYSIS OF THE SCIENTIFIC PRODUCTION OF CEMENTITIOUS COMPOSITES WITH RECYCLED POLYMERIC MATERIALS

Concrete that is a cementitious composite is the second most used material in the world, however, for its production there is a need to use non-renewable natural resources. On the other hand, there is an increasing increase in polymeric waste that could be used as an aggregate and thus mitigate the use and non-renewable natural resources. In this context, there is an increasing amount of research on this topic. However, despite the large amount of data in this field, there is no consensus on the effect of adding or replacing polymeric residues in cementitious composites on durability, structure and properties. In this sense, this work aims to make a bibliometric survey on cementitious composites with the addition of polymeric residues in order to know where these studies are most advanced, which are the most productive research groups. It was conducted research on the worldwide scientific production on cement composite with recycled polymeric constituents. Conventional concrete (CC) and self-compacting concrete (SCC) were selected as bibliometric indicators. It aimed to analyze the number of articles and other studies published in journals five important databases on the theme portland cement composite. Three thousand three hundred one articles were analyzed for CC and 137 articles for SCC, produced internationally, and indexed in the Scopus database. These data were used in the VOSviewer® software to build and visualize bibliometric networks. In quantitative terms, the journals, authors, years, and institutions that publish the most on the research topic were identified. The analysis of the data generated confirms that the field of concrete with polymeric waste has significant attention in the scientific field, mainly in countries such as India, the United States, China, and Iraq that contribute significantly to this field of research.

2.3 Introduction

2.3.1 Contextualization of bibliometric review and data analysis

The bibliometrics technique was developed at the beginning of the century to supply the need for studies and evaluation of communication activities and scientific production. Bibliometrics can be understood as a quantitative and statistical technique to measure production indicators and the rapid dissemination of scientific knowledge [34].

Bibliometrics uses the application of statistical and mathematical techniques to describe characteristics as well as other means of communication. It is an analysis of quantitative information that was originally called "statistical bibliography" by Hulme in 1923. In 1934, Otlet created the term bibliometrics in his "Traité de Documentation" [35].

With the advent of technology and data storage, scientific research has undergone and is undergoing deep transformations that require the adoption of new research instruments and more careful data treatment refinements [36]. It is important to understand this new reality, its dynamics, and complexity because that is the only way to produce consistent results through the indicators in a detailed and systematic way. The current technological and scientific scenario is dynamic and has countless sources of information, which in turn vary greatly in the credibility and quality of the data reported [37].

The use of a tool that optimizes searches for scientific publications in the initial study periods is extremely important so that researchers do not reproduce published results, thus saving time. Bibliometrics provides a method of analysis that provides an overview of a given topic with several relevant indicators [38]. Bibliometrics measures the contribution of scientific knowledge, based on scientific publications in certain areas of knowledge. These data can be used to map current trends, as well as to identify topics for new research [39].

In short, bibliometrics is a network search engine that uses quantitative and statistical techniques to survey production rates in certain scientific areas, thus providing a means of quantifying data to contribute to scientific knowledge. With this, it is possible to obtain information such as: who are the main authors, the years, the institutions, the countries, and the areas that are related to the topic of interest [40]. The bibliometric analysis method can help in the evaluation of research data in publications and their citations, which are increasingly used in various research evaluation processes. The popularity of bibliometrics has increased in recent years due to the development of comprehensive and available databases, for example Scopus, Dimensions and web of Science. Scientists making use of these databases can correlate the numbers indicators with possible research trends [41]. The high degree of bibliometric procession questions the use of expensive and time-consuming peer review processes in certain decision-making processes such as classifications of institutions, disciplines that are related to peer review assessments. A comparison of the use of bibliometrics and peer review is demonstrated by [42]. In their studies, they show that the use of bibliometrics reaches results similar or even superior to the peer review processes, which is a time-consuming and expensive process. The authors demonstrate that article citations are strongly linked to peer review assessments at the level of institutions and disciplines and processes that involve data.

2.3.1.1 Plastic waste

Plastic is one of the greatest innovations of the 20th century and a ubiquitous material worldwide. Recently, in the last few decades, the consumption of plastic material has grown significantly, which has led to a considerable accumulation of plastic waste. As a result, it is possible to find plastic products in all ecosystems on the planet [43].

In the last decades, a large amount of non-degradable waste, particularly in the form of plastic waste (PW), has promoted serious environmental challenges. Furthermore, PW is considered one of the most dangerous forms of pollution, due to their chemical and physical characteristics and long life cycle [44–47]. In 2017, 348 million tons of plastic were produced worldwide, of which 64.4 million tons in the European continent alone. Approximately 31.1% (108 million tons) of this total was recycled and disposed of as waste 27.2% (95 million tons) [48].

In 2018, over 400 million tons (Mt) of plastics were produced, among which 164 Mt were destined for packaging, 36% of the total [49]. The packaging of industrialized products represents approximately one third of all used plastics, of which close to 40% is destined for landfill. Currently the amount of plastics actually measured in the oceans represents less than 1% of the 150 Mt estimated to have been released into the oceans over time [49]. Plastic pollution has grown rapidly in all ecosystems in the world, in the oceans plastics pose a major threat to marine life [50]. About 10% by mass of urban waste is made up of plastic, territorially the five most polluting countries of plastic are: China, Indonesia, Philippines, Vietnam, Sri Lanka these alone contribute 56% of the global plastic waste [49]. Brazil, according to data from the World Bank, is the 4th largest producer of plastic waste in the world, with 11.3 million tons. Still, only 145 thousand tons (1.28%) are effectively recycled, according to Table 2-1 that summarizes data on the relationship between waste production and recycling in several countries [51].

Table 2-1: Production and recycling of plastic in the world in tons [51]

Country	Total plastic waste generated	Total incinerated	Total recycled	Percentage ratio of production and recycling (%)
United States	70.782.577	9.060.170	24.490.772	34,6
China	54.740.659	11.988.226	12.000.331	21,9
India	19.311.663	14.544	1.105.677	5,7
Brazil	11.355.220	0	145.043	1,3
Indonesia	9.885.081	0	362.070	3,7
Russia	8.948.132	0	320.088	3,6
Germany	8.286.827	4.876.027	3.143.700	37,9
United Kingdom	7.994.284	2.620.394	2.513.856	31,5
Japan	7.146.514	6.642.428	405.834	5,7
Canada	6.696.763	207.3540	1.423.139	21,3

Because the polymer has a low rate of degradation and is naturally bulky, landfills cannot be considered an ecological alternative for the disposal of PW [52]. The decomposition time for wastes, including plastics and rubber that have a polymeric nature, can reach more than 400 years. The bulky waste of the PW can obstruct groundwater flows and prevent the growth of tree roots, among other problems. The PW can contaminate the water and the soil when they are mixed with the rainwater. With this, they disperse in the environment where there are toxic elements, such as lead and cadmium [52].

2.3.1.2 Concrete with plastic waste

Concrete after drinking water is the most consumed material daily in the world, so research aimed at incorporating recycled waste into this cementitious composite is of great social value [53]. To reduce the influence of PW on the environment in terms of energy consumption, natural resources, waste disposal, global warming, and environmental pollution, recycling of PW can represent one of the best solutions [54, 55]. The use of PW as an aggregate in concrete is investigated in several studies that generally recommend its use for non-structural functions, in addition to describing methods of obtaining and preparing waste for use in concrete [52–54, 56–60].

In practical applications in civil engineering, the recycling of plastic waste can be incorporated as a base for cementitious material, such as in mixtures of mortar and concrete. Due to ecological and economic advantages, these plastic waste can replace or work together with the traditional natural aggregates of concrete [61].

Concretes with plastic waste, in general, have low specific mass and are ideal for applications of non-structural elements and concrete asphalt pavements [55]. Many studies prove the applicability of concrete with plastic waste in different situations. There are several studies on types of polymeric wastes used as concrete aggregates, for example, [61] using polyvinyl chloride (PVC) waste tubes, [45, 62] polyethylene terephthalate (PET) waste, [63] waste from high-density polyethylene (HDPE), [64] expanded polystyrene foam (EPS) waste, [65] recycled thermoplastic polystyrene waste, and [66] polypropylene fiber.

2.3.1.3 Self-compacting concrete and polymeric waste

Among the cementitious composites, SCC has been gaining prominence for its unique qualities in the fresh state that allows its use in densely armed structures and difficult to access. Thus, its use is indicated for medium to large construction and for reinforcement and revitalization of structures [57, 67].

Self-compacting concrete (SCC) fits into a special type of concrete that has the characteristic of self-compacting, that is, due to its own weight, it can flow and fill the mold, without requiring external vibration [67–69]. However, to obtain such a result, the SCC needs to have cohesion and fluidity in equilibrium such that segregation and exudation of its constituent materials do not occur [70]. To obtain adequate workability, the SCC traces often contain the addition of superplasticizer additives, which reduce the demand for mixing water and fine material (silica fume, stone powder, fly ash, and blast furnace slag) that improve stability and granulometric packing of concrete [71]. Viscosity modifying additive can also be used along with fine materials to achieve adequate cohesion and resistance to segregation [72, 73]. SCC is frequently used to incorporate plastic waste, thus generating new characteristics for conventional self-compacting concrete. However, it is necessary to take a due care to maintain the main characteristics of SCC, which in the fresh state are fluidity, stability, and homogeneity, and in the hardened state are better mechanical properties and good durability [74].

Given the lack of data and studies that compile the number of studies related to the concrete theme with polymeric waste, and knowing the great importance of the research that shows the current scenario related to the theme, we tried to carry out this study through bibliometric indicators linked to the concrete with the polymeric waste theme, through a multidisciplinary and methodological approach. The use of bibliometric indicators requires users to have a comprehensive view of the area of specific knowledge and the limitations inherent to each segment.

In this sense, this work aims to make a bibliometric survey on cementitious composites with the addition of polymeric residues in order to know where these studies are more advanced, which are the most productive research groups and the number of studies that are being done. Another important point is that this type of research seeks to avoid bias or biased research that compromises the interpretation of data for the formation of standards and best practice procedures. It even helps in the definitive exclusion of certain material in some field of possible use.

2.4 Methodology

The research developed is based primarily on the choice of the engineering database that generates significant results in the search for documents related to the topic. The database chosen should initially cover much of the literature related to the topic: CC and SCC with polymeric waste. To choose the database, string refinement steps (with adjustments to keywords and Boolean operators) were carried out in a search that was inserted in 5 different databases.

Besides quantifying criteria concerning the coverage of articles, books, and works, the selection of the database used in the bibliometric research took into account available tools to analyze the results, such as the generation of graphs and diagrams available on the database platform. Initially, several search strings were developed for five different databases: Scopus, Web of Science, Scielo, Engineering Village, and Science Direct. These strings were refined and used in the same way in all selected databases. The refinement considered the number of results generated in each search.

After choosing the database and defining the search string, the database search started working in its search fields. Right from the start, the options for "documents" and search in "titles," "keywords," and "abstracts" were selected. The period considered, both for searching with CC and for SCC, was the entire period of data in the selected database, so a period of predefined years was not limited.

To assist in the analysis of data extracted from the selected database, the VOS viewer software was used. The VOS viewer software is a tool developed for the construction and visualization of a bibliometric network [75]. These networks can contain data such as the names of newspapers, researchers, publications, countries and can be constructed considering criteria such as citation, academic production, co-authorship and others [75]. In this study, VOS viewer was used to analyze the keywords searched and other terms searched.

To do so, the same search conducted in the Scopus database was used. Figure 2-1 shows a flow chart that summarizes the methodology adopted by this study, as well as the phases to get to the results.

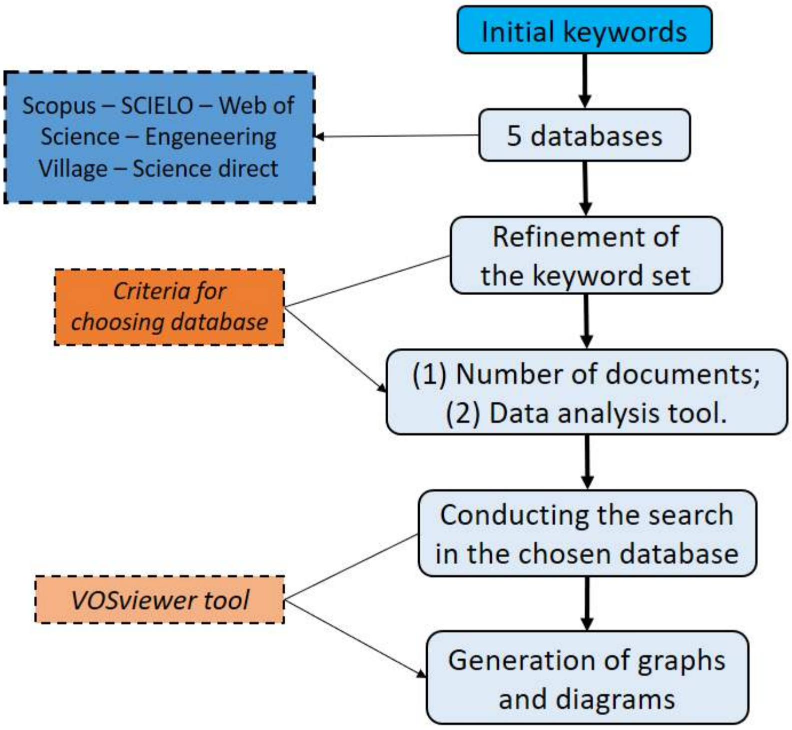

Figure 2-1: Flowchart of the adopted search methodology (AUTHOR, 2022).

After the generation of graphs and diagrams with bibliometric data, the results were analyzed seeking to link the historical, political and economic events that involve the development of the use of concrete with polymeric residues and that justify exactly the behavior of the data in certain analyzed periods. Data analyzes were performed in related ways, that is, the results obtained in certain analyzes were used to confirm trends observed in other results.

2.5 Results and discussion

2.5.1 Choice of database

The Figure 2-2 shows the results of searches for databases for the CC theme with polymeric waste. And the Figure 2-3 shows the results of the search carried out for the SCC with polymeric waste.

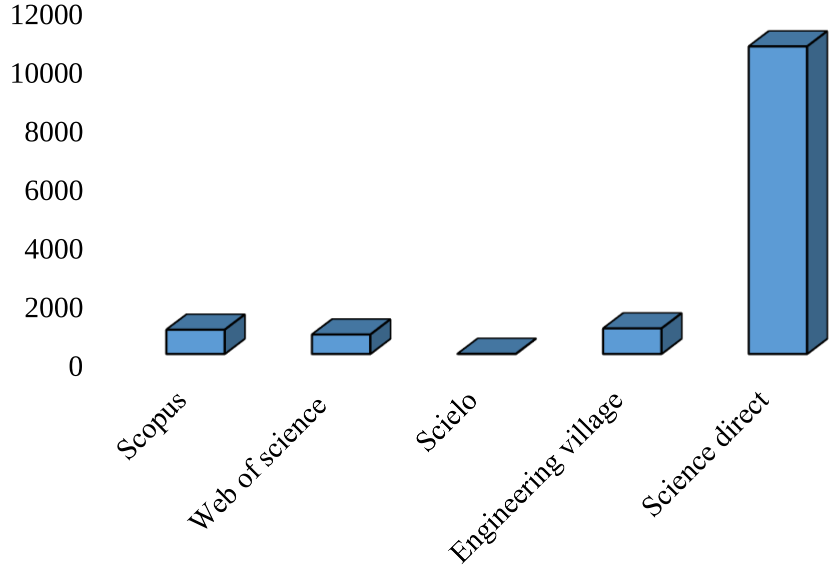

Figure 2-2: Number of articles searching with the string: concrete AND (plastic* OR polymer* OR RUBBER*) (AUTHOR, 2022).

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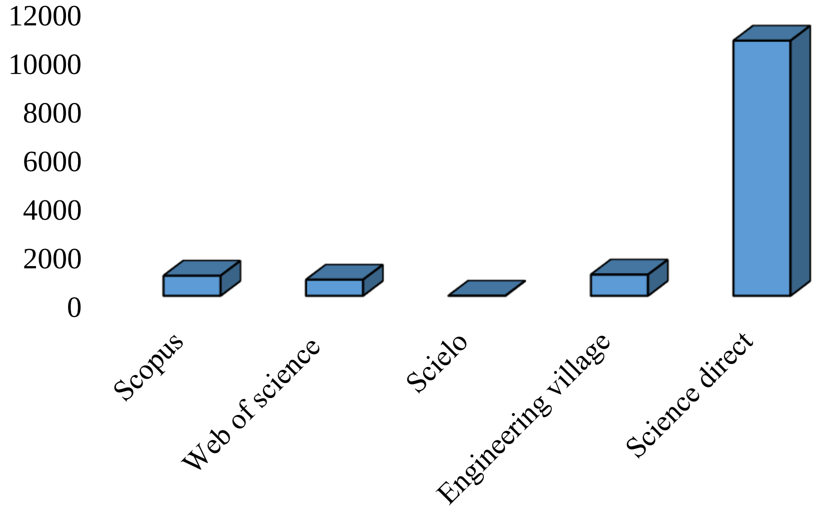


Figure 2-3: Number of articles searching with the string: self AND compacting AND concrete AND (plastic* OR polymer* OR rubber) (AUTHOR, 2022).

As can be seen in Figure 2-2, the bases that generated the highest results were Science Direct, Scopus, and Engineering Village, respectively, for CC. For SCC, the greatest results were found in Science Direct and Engineering Village and Scopus, respectively, as can be seen in Figure 2-3.

Based on this research, we then chose to choose the Scopus database, as it provides a considerable number of results for the research carried out and because it is a database that has a large part of relevant journals related to engineering, besides offering computational resources of data analysis that facilitate the organization of collected data and discussions of results. Resources similar to these are not provided by the Science Direct database that generated the most results.

After the database selection step, a further refinement was made to the search string. It was decided to introduce the keywords waste and residue to restrict the field of research to plastic waste or polymers. Figure 2-4 shows the results for both CC and SCC.

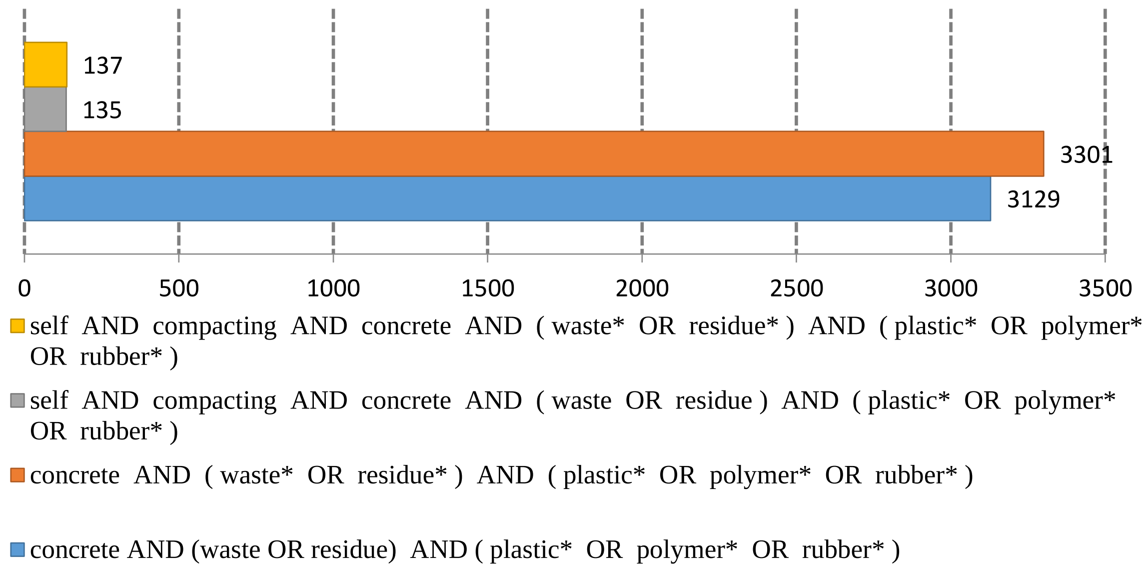


Figure 2-4: Document numbers in search at Scopus (AUTHOR, 2022).

From this stage, to conduct the research, only the Scopus database was used. For this purpose, the CC research was concentrated in the period from 1954 to 2021, as this was the deadline provided by the database, knowing that this study seeks to be developed in the longest possible coverage time. For SCC, filters from 2003 to 2021 were used, which is also the period provided by the database. Authors linked to institutions of all nationalities were considered. Three thousand one hundred twenty-nine peer-reviewed scientific articles were selected, as well as material such as books, theses, and dissertations related to the CC theme with polymeric waste, and 136 SCC documents with polymeric waste.

Figure 2-5 and Figure 2-6 show the results for the search for the most relevant keywords related to the theme of this study.

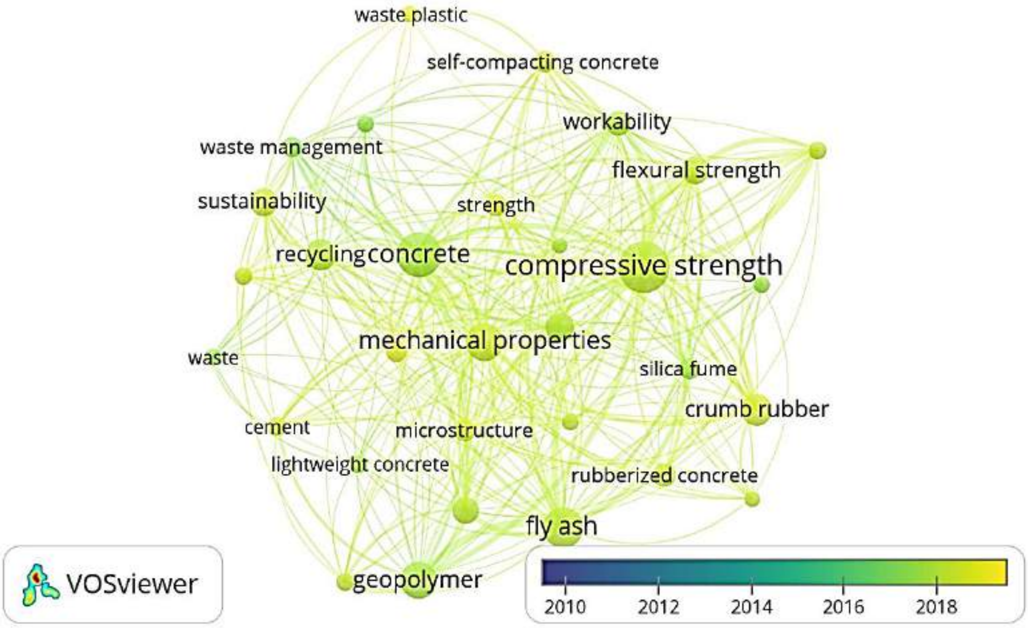


Figure 2-5: Relevant keywords on cement composite with recycled polymeric constituents for CC from 2010 to 2021 (AUTHOR, 2020).

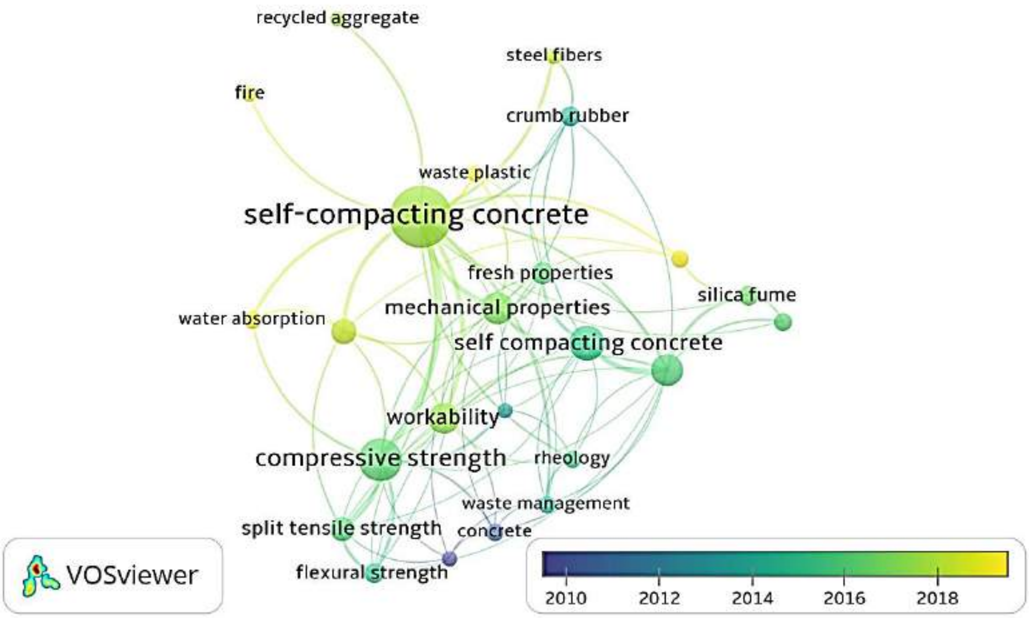
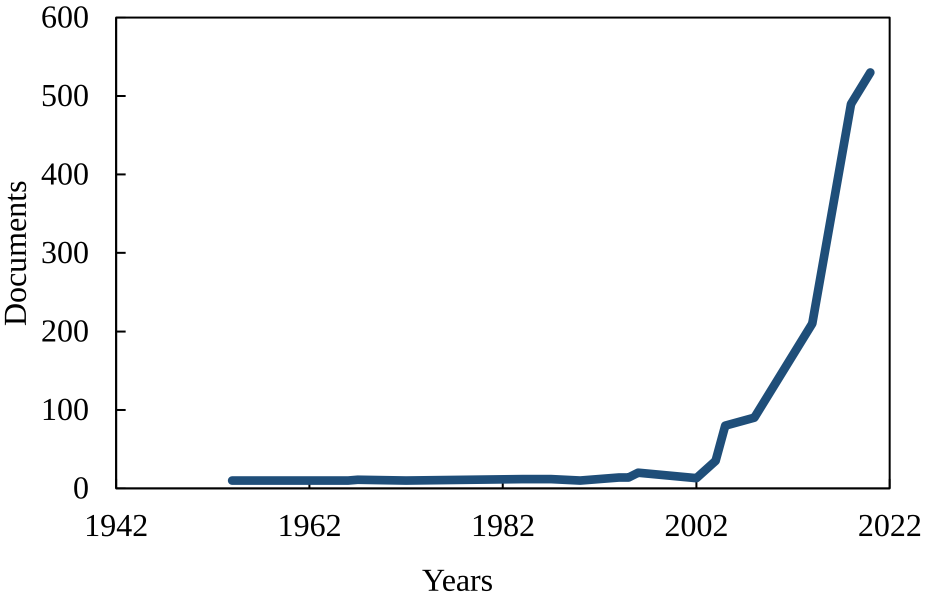


Figure 2-6: Relevant keywords on cement composite with recycled polymeric constituents for SCC from 2010 to 2021 (AUTHOR, 2022).

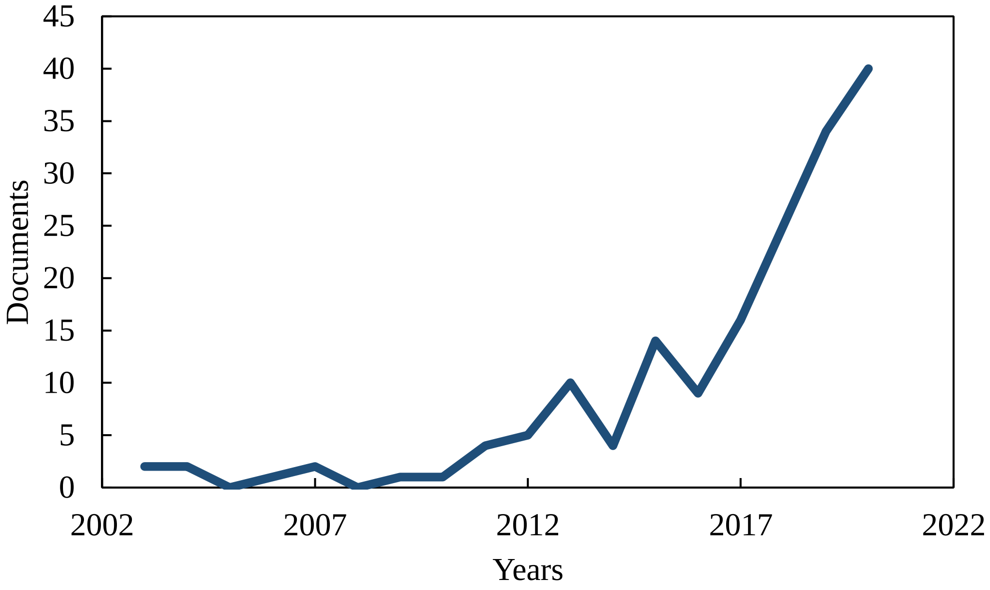
The keywords are organized to show the connection between them and in which period 2010 to 2021 the authors most cited them. In general, for CC, the keywords are recent and were mostly cited between 2016 to 2021. For SCC, there are many current cited keywords between 2019 and 2021. But others had their moments of relevance about 8 to 10 years ago, as is the case with flexural strength, waste management, crumb rubber.

2.5.2 Documents per year

The Figure 2-7a and Figure 2-7b shows the number of documents published per year related to CC and SCC with polymeric waste [76].



Conventional concrete (a)



Self-compacting concrete (b)

Figure 2-7: Documents per year on cement composite with recycled polymeric constituents for CC and SCC with polymeric waste (AUTHOR, 2022).

The number of documents per year has grown sharply since the beginning of the 21st century, around 2002. In the years 2008 to 2014, the scientific production related to the theme, exceeds the number of 100 documents per year, and from 2014 it exceeds 200 documents per year. The sharp growth continues until the moment when the number of documents approaches 500 per year.

The production of SCC with waste, as shown in Figure 2-7b, has grown significantly since the last decade, reaching close to 35 documents in 2021. The number of documents related to the SCC theme represents approximately 5 to 10% of CC production in the period from 2019 to 2021, Figure 2-7a. SCC is a special type of concrete that was developed in the 1980s in Japan by researcher Okamura and his team [69]. Nonetheless, recycled plastic materials started to be adopted more frequently as an aggregate for both CC and SCC in the early 21st century.

The marked production of scientific studies related to the concrete theme with polymeric waste can be understood as a consequence of public policies concerning the environment that had been gradually discussed and adopted by countries in world conferences on the environment [70]. The international events that propelled the constituted powers of the countries to take mitigating measures regarding the environment follow a historical line that begins in the 70s: The Club of Rome (1972), The Stockholm Conference on Environment (1972), A UN World Commission on Environment and Development (CMMAD)(1983/1987), The United Nations Conference on Environment and Development (UNCED)(1992), The Johannesburg World Summit on Sustainable Development (2002)[77].

These events discuss issues related to the environmental crisis and are gaining strength and impacting the goals of the government in search of sustainable and no longer exploratory and inconsequential development [77]. At this scenario, environmental public policies are the main stage of international economic-industrial expansion and are aimed at reducing harmful human intervention in the environment, which generates, among other problems: global warming, acid rain, contamination of water sources, desertification, deforestation, excessive CO2 emission, species extinction, melting of polar ice caps, depletion of natural resources.

The growth due to the interest in the concrete theme with plastic waste does not happen by chance, particularly in the early 2000s, but it follows a worldwide trend of preserving the environment and with measures that impose obligations for recycling and reuse of waste. With the development of society, there is a need for infrastructure development [78]. New characteristics are required for concrete, and new aggregates are studied to decrease the demand for natural aggregates that are limited in nature [79].

2.5.3 Documents by author

The Figure 2-8a and Figure 2-8b lists the main authors who publish documents related to CC and SCC with polymeric waste [76].

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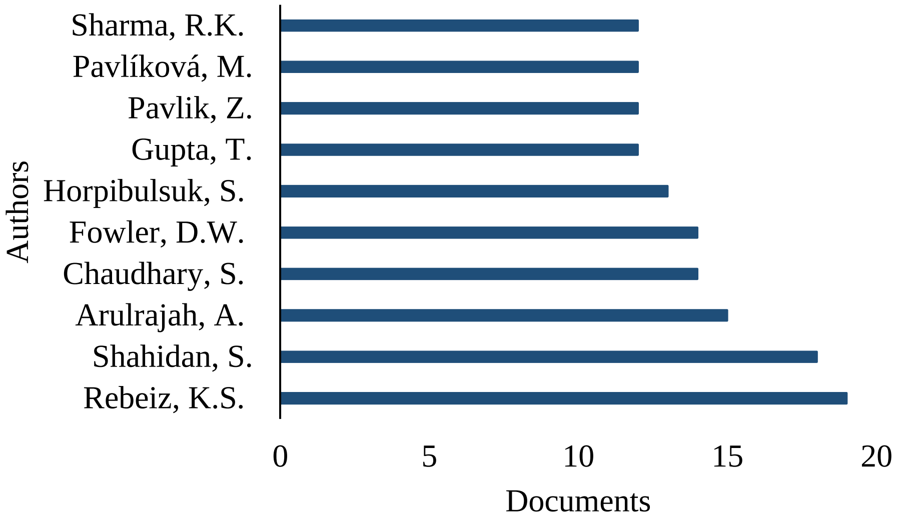
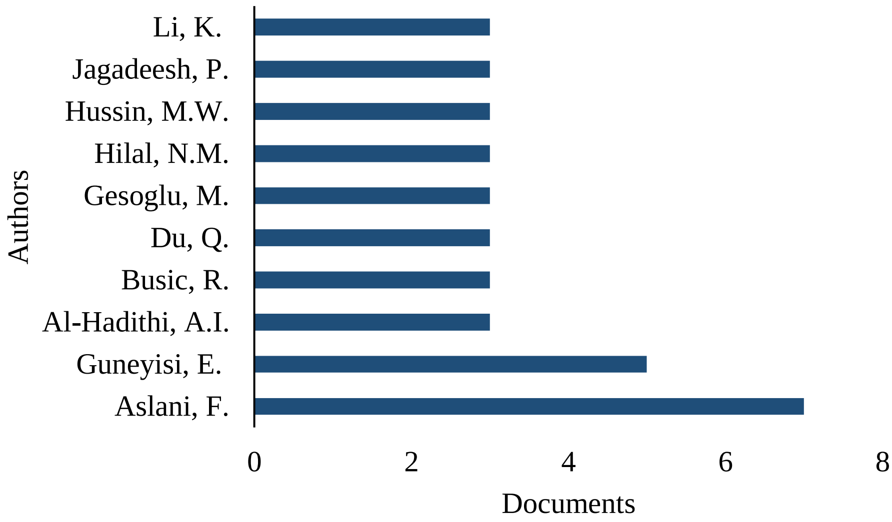


Conventional concrete (a)



Self-Compacting Concrete (b)

Figure 2-8: Documents by author for cement composite with recycled polymeric constituents on CC and SCC (AUTHOR, 2022).

Figure 2-9 shows the results for searching the VOSviewer® Software for the most relevant authors linked to the theme of this study.

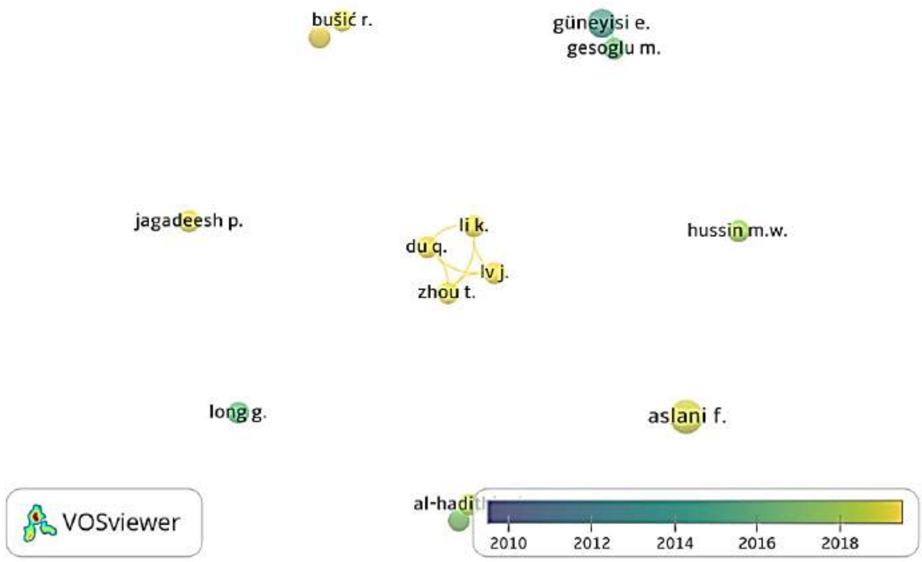


Figure 2-9: Relevant authors on SCC with polymeric residues from 2010 to 2019 (AUTHOR, 2022).

2.5.4 *Documents by journal*

Figure 2-10 list the main journals that published documents related to CC and SCC with polymeric waste [76].

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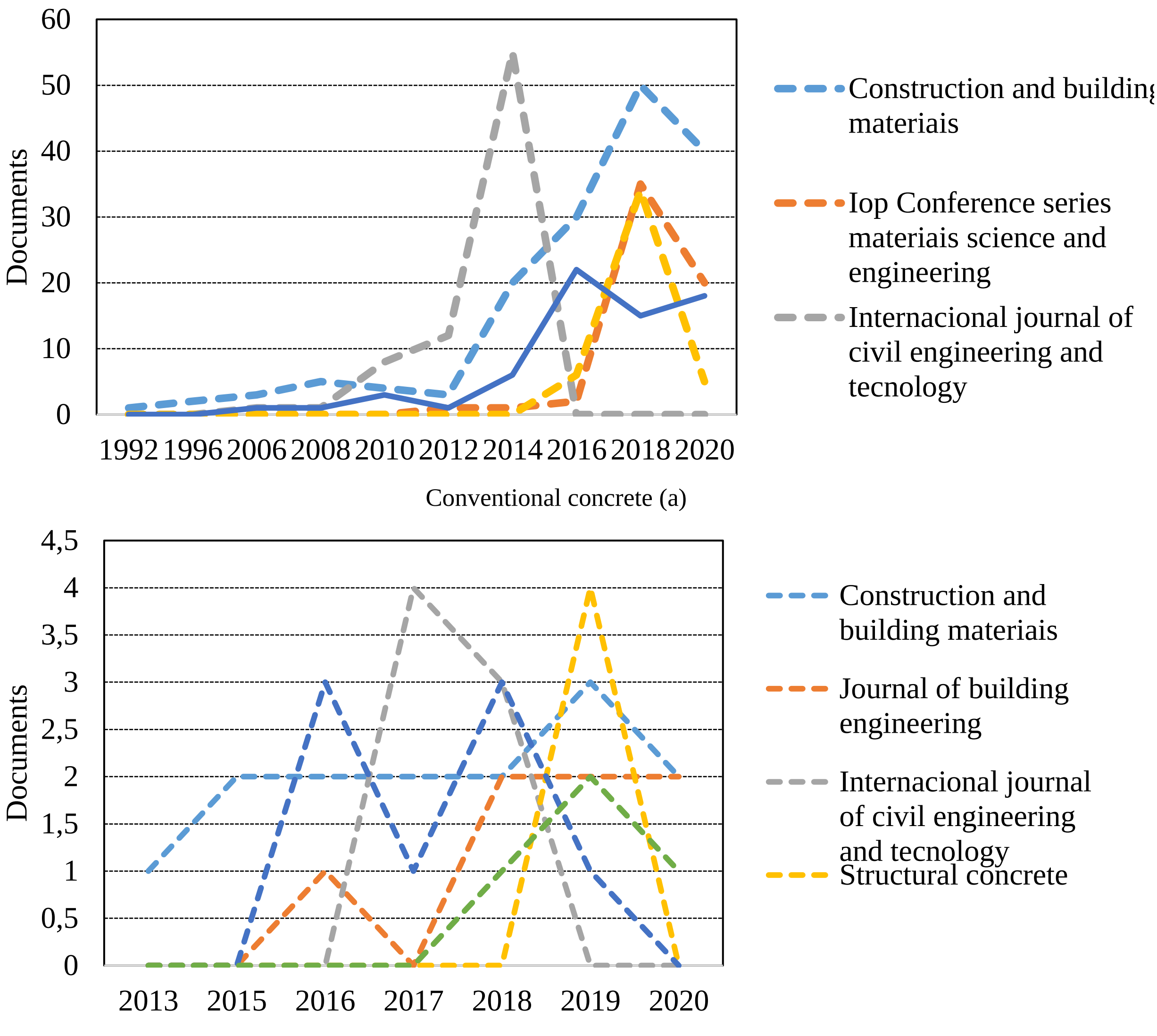


Figure 2-10: Documents by journals for cement composite with recycled polymeric constituents on CC and SCC (AUTHOR, 2022).

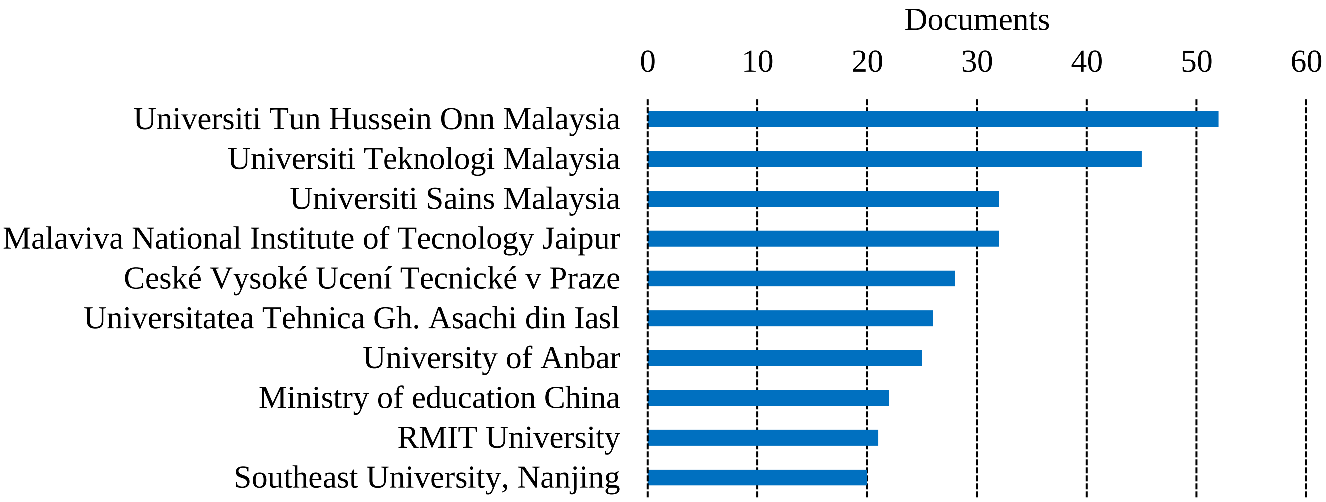
Through the results, it is possible to notice that the main journals are: Construction and Building Materials, Advanced Materials Research, Iop Conference Series Science and Engineering, Journal of Cleaner Production and International Journal of Civil Engineering and Technology. The following should be highlighted: Construction and Building Materials and Advanced Materials Research, which contributed with more than 40 documents per year in the decade from 2010 to 2020. The journals Iop Conference Series Science and Engineering and Journal of Cleaner Production and International Journal of Civil Engineering and Technology are in the range of 20 to 40 documents produced per year.

Publications, in general, appear to be increasing in most newspapers. The apparent decrease shown in the 2019 to 2021 biennium should not be taken into account, as the publications for that period were probably still being updated in the database. In Figure 2-10, the results show that the journals that most publish documents related to the SCC theme with polymeric waste are: Construction and Building Materials, Internacional Journal of Civil Engineering and Technology, Journal of Cleaner Production, Journal of Building Engineering, Materials and Structural Concrete. The Construction and Building Materials journal, which kept the number of publications constant from 2013 to 2021, should be highlighted. On average, journals publish around 2 to 3 documents related to the SCC theme with polymeric waste per year.

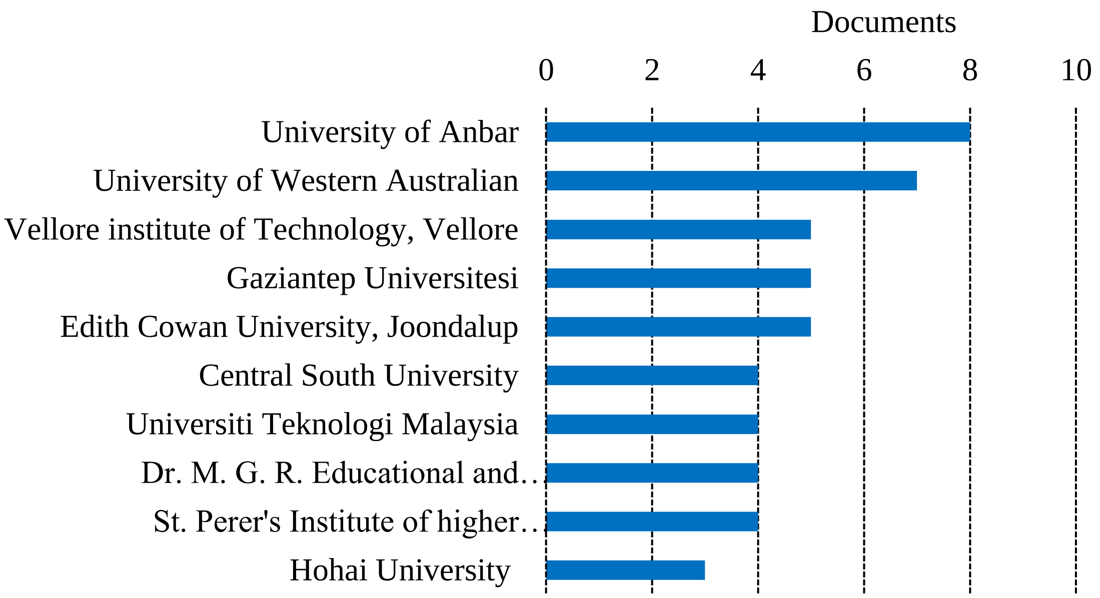
Some considerations about the journal's origin can be made, considering that the main journals listed in Figure 2-10 are located in Europe. The European continent is consistent with the international trend, given that the European Union seeks to expand its collaborations with countries on the Asian continent, especially China . The European Union does not appear to be seriously affected by internal and external economic problems. Investment in research comes largely from around 36% of the public sector [80].

2.5.5 Documents by institution

The Figure 2-11 lists the main institutions that publish documents related to concrete and SCC with polymeric waste [76].



Conventional concrete (a)



Self-compacting concrete (b)

Figure 2-11: Documents by institutions for cement composite with recycled polymeric constituents on CC and SCC (AUTHOR, 2022).

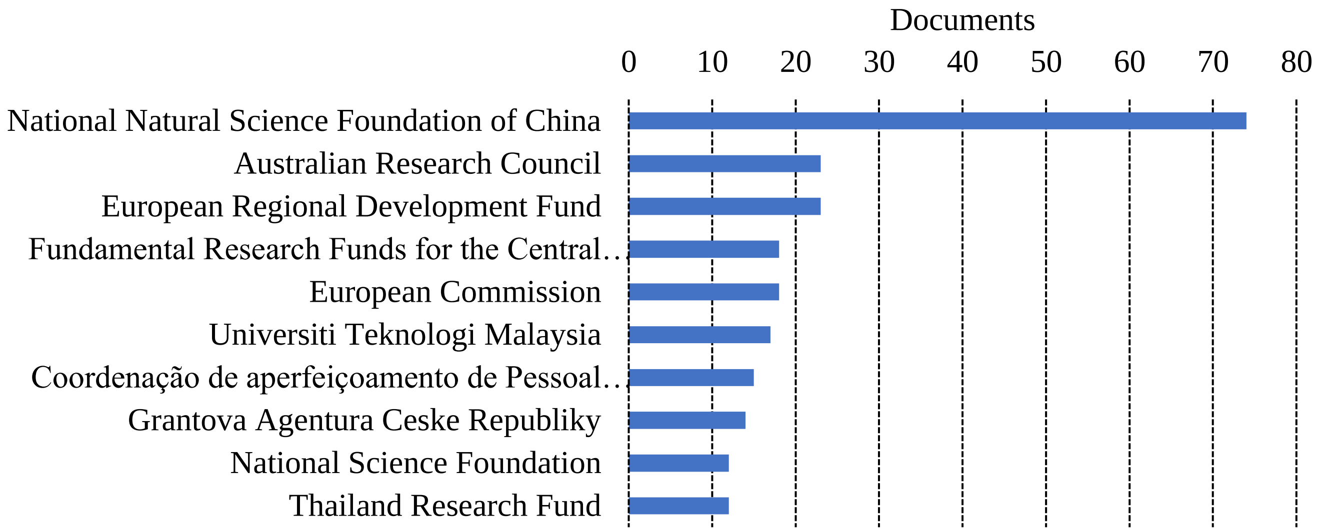
The main institutions linked to the production of documents related to the concrete theme with polymeric waste are: Universiti Tun Hussein Onn Malaysia (Malasia), Universiti Teknologi Malaysia (Malasia), Universiti Sains Malaysia (Malasia), Malaviya National Institute of Technology Jaipur (India), Ceské vysoké ucení technické v Praze (Czech Republic), Universitatea Tehnica Gh. Asachi din IasI (Romania), University Of Anbar, Ministry of Education China (China), RMIT University (Australia), Southeast University, Nanjing (China). The institutions that appear as the largest producers of documents related to concrete with polymeric waste are linked to the countries with the greatest scientific production related to the topic on concrete with polymeric waste. Therefore, it is consistent that these institutions are highlighted.

It should be noted that the Asian continent, represented in large percentages by Malaysia and China, respectively, are the countries that most produce research on the subject in question. This is certainly mostly due to infrastructure investments made in these countries over the past decade. The Chinese and Indian economies, in particular, since the late 1990s have shown annual growth above 8% [81]. Malaysia, which is located in Southeast Asia, is an aspiring Asian tiger country (Hong Kong, South Korea, Singapore, Taiwan) since its foreign investments come from the Asian tigers themselves that drive its development [82]. This group of countries of marked economic development identified the incentive for research and development as the main factor in economic development [80].

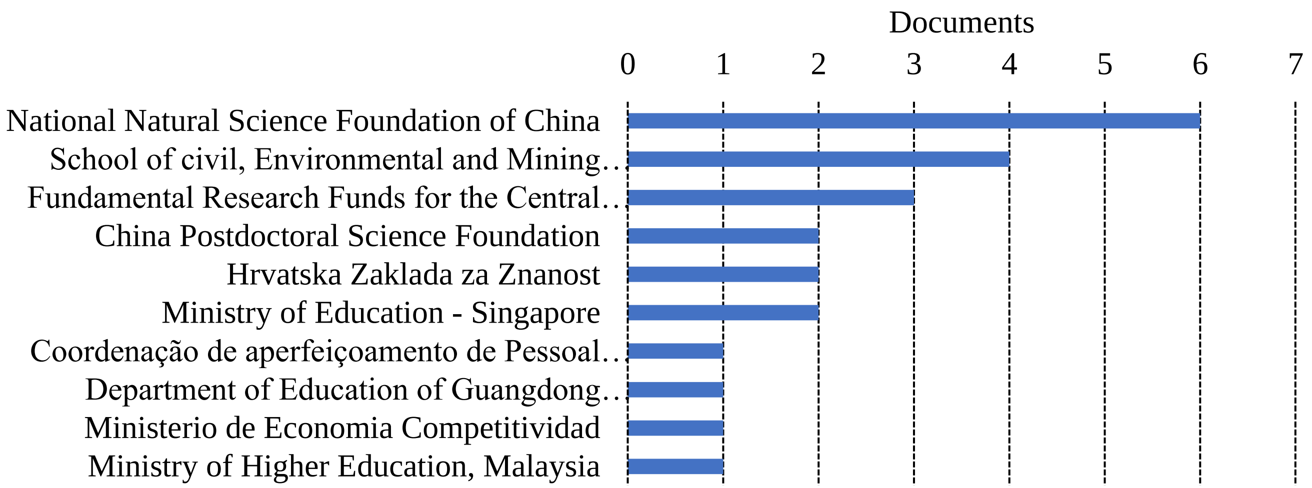
Regarding SCC with polymeric waste, the main institutions are: University Of Anbar (Iraq), University of Western Australia (Australia), Vellore Institute of Technology (India), Vellore Gaziantep Üniversitesi (Turkey), Edith Cowan University (Australia), Joondalup Central South University (Australia), Universiti Teknologi Malaysia (Malasia), Dr. M.G.R Educational and Research Institute (India). St. Peter's Institute of Higher Education and Research Hohai University (India). The classification of institutions is certainly linked to the investment of the respective countries of origin of each institution. For example, India, China, Iraq, and Australia are the countries that produce the most scientific documents related to the topic. Therefore, the institutions located in their territories are more prominent in this field of study, as with institutions linked to the research of CC with polymeric waste.

2.5.6 Main sponsors/financiers

The Figure 2-12 lists the main sponsors/financiers that publish documents related to CC and SCC with polymeric waste [76].



Conventional concrete (a)



Self-compacting concrete (b)

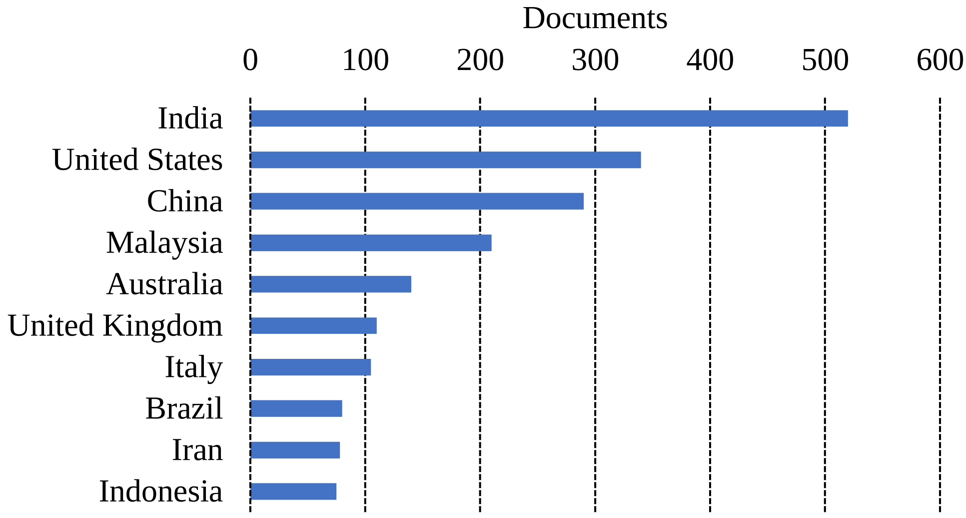
Figure 2-12: Documents by sponsors / financiers for cement composite with recycled polymeric constituents on CC and SCC (AUTHOR, 2022).

The main funding agencies for studies related to concrete with polymeric waste are: National Natural Science Foundation of China, Australian Research Council, European Regional Development Fund, Fundamental Research Funds for the Central Universities, European Commission, Universiti Teknologi Malaysia, Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES), Grantova agentura ceske republiky, National Science Foundation (NSF), Thailand Research Fund. The main sponsors/financiers of research are largely located in Asia, with emphasis on China, which has been consistently investing in research since 1990. As a comparative measure, China's economy has grown between 9% to 10% in recent years, while investments in research have increased at the rate of 12% [80].

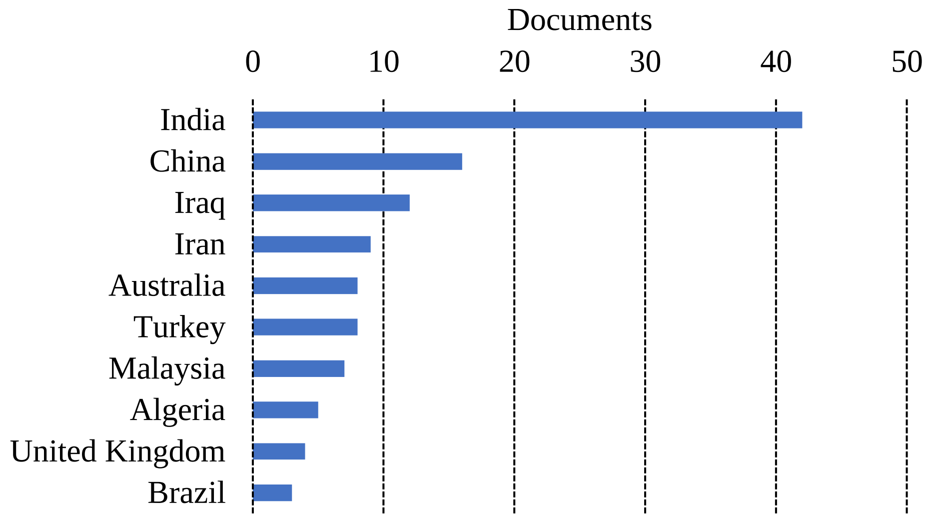
For SCC with polymeric waste, the main funding agencies are: National Natural Science Foundation of China, School of Civil Environmental and Mining, Engineering University of Adelaide, Fundamental Research Funds for the Central Universities, China Postdoctoral Science Foundation, Hrvatska Zaklada za Znanost, Ministry of Education – Singapore, Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES), Department of Education of Guangdong Province, Ministerio de Economia y Competitividad, Ministry of Higher Education, Malaysia. For both CC and SCC, China's funding agency, the National Natural Science Foundation of China, appears as the main sponsor. We highlight the School of Civil Environmental and Mining and the Engineering University of Adelaide, who contributed with 4 and 3 studies, respectively. CAPES appears funding only 1 study on the topic. It is worth mentioning that the growth in research and development in Asian countries is a reflection of the rapid economic advance, population growth, and the consequent training of a generation of trained professionals [80]. Between 2003 and 2008, the number of researchers in Taiwan, China, Singapore, and South Korea grew by approximately 16%. In contrast, in the USA, the number of young researchers decreased from 51% to 49%. As a comparison with the publication of articles, in Asia, in the last decade, there was an increase of 9% per year, while in the USA and the European Union only 1% [83].

2.5.7 Documents by countries and territories

The Figure 2-13 lists the main countries that publish documents related to CC and SCC with polymeric waste [76].



Conventional concrete (a)



Self-compacting Concrete (b)

Figure 2-13: Documents by country or territory for cement composite with recycled polymeric constituents on CC and SCC (AUTHOR, 2022).

The countries India, USA, China, Malaysia, and Australia produce a large number of studies related to concrete with plastic waste. It is worth highlighting India, which reached the mark of 500 documents in the surveyed period, representing the sum of several countries together. Both developed countries (USA and China), as well as countries that are in the process of development (India, Malaysia, and Brazil), need large volumes of concrete to develop. It is possible to draw a parallel between investment in infrastructure and the consumption of concrete [79]. It is interesting to make a correlation between the countries that produce the most polymeric wastes and the countries that produce the most studies on recycling these wastes in the form of aggregate for concrete.

It can be seen in Table 1 that the largest producers of waste (USA, China, India) are also one of the largest producers of studies related to CC and SCC with polymeric waste.

The World Economic Forum 2017/18 [84] released a classification of the percentage of accumulated expense on infrastructure investments in the last five years that follow the following sequence: Asia-Pacific 61%; USA and Canada 12%; Western Europe 11%; Latin America 5%; Central and Eastern Europe 5%; Middle East 4%, and Africa 2%. These data can be used to understand the massive use of concrete and recycled material, mainly by Asian and Pacific countries, as infrastructure investment involves the investment in various aspects of society, such as sustainable development combined with scientific research [84].

Regarding the SCC with polymeric waste, Asian countries are also highlighted in terms of the number of documents published, as seen in Figure 13. The Asian countries represented by India, China, and the Middle East Iraq and Iran, as well as Oceania, Australia represent regions of the world where there is a greater incentive to research and development [84]. As in China, where the government facilitates tax deductions for research and development and creates local awards with cash incentives for authors of impact articles. The government encourages the transfer of scientific knowledge to the industrial and commercial production sector to obtain an economic return [80].

In the case of the USA, participation in research in general for the scientific public has decreased. One reason is that the government's participation in this type of research and development investment has been decreasing, and the sector that most felt this slowdown was the Department of Defense [80]. Although government spending is decreasing, industries, private universities, and NGOs continue to make their investments and try to compensate for the reduction in public incentives. For this reason, the USA, in general, is still one of the main producers of research [83]. Although the USA does not invest at the pace of the Asian countries mentioned, it is still the largest research power in the world. Something that can be attributed to the fact that Americans do not appear among the main research developers in the area of SCC with plastic waste is that the USA focuses its research investments as a business product, mainly in the field of engineering. Making development and improvements in products and services that remain as a business segregate and that do not contribute significantly to the scientific community, concerning the dissemination of knowledge as belonging to the public [85].

Figure 2-14 and Figure 2-15 show the results for the search for the most relevant countries linked to the theme of this study.

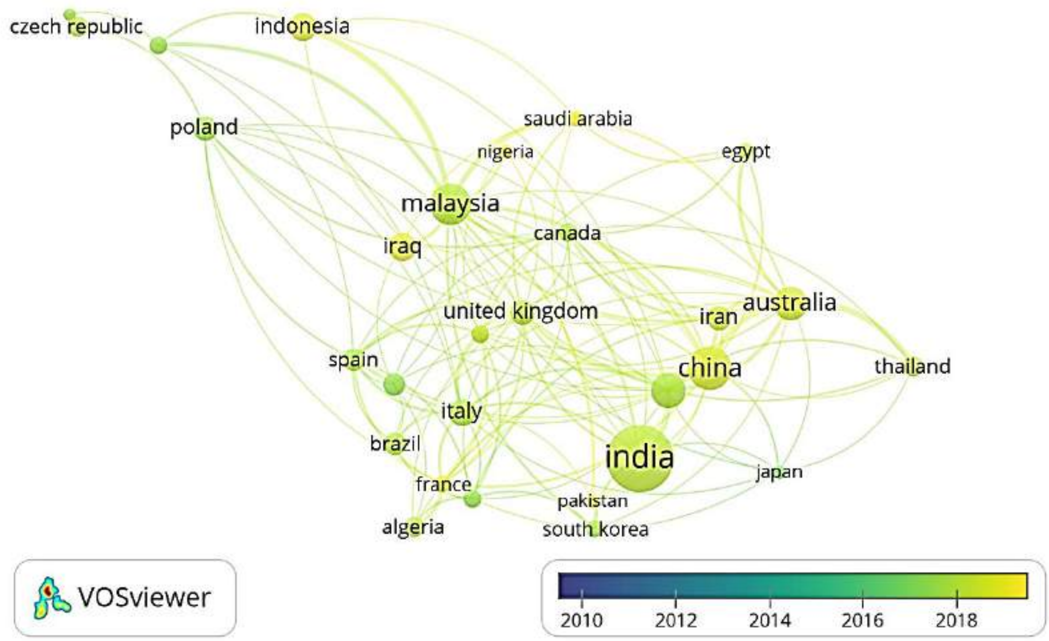


Figure 2-14: Relevant countries on CC with polymeric wastes from 2010 to 2021.

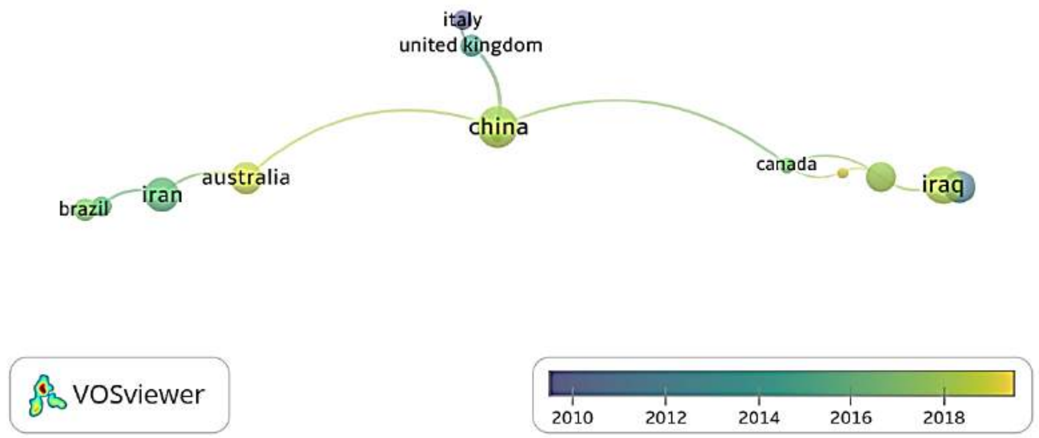
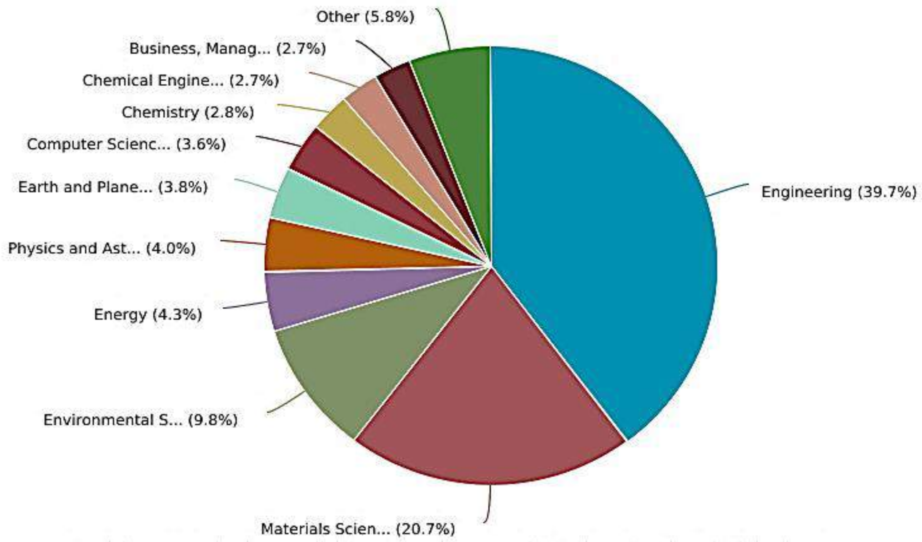


Figure 2-15: Relevant countries on SCC with polymeric waste period from 2010 to 2021 (AUTHOR, 2022).

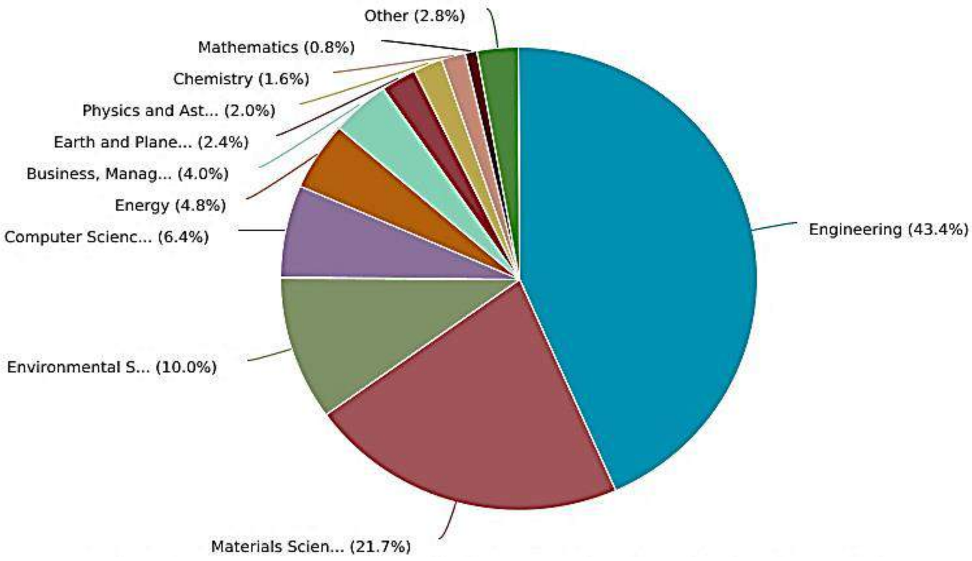
There is a link or collaboration between research carried out in the main countries for both CC and SCC. The results shown in Figure 2-14 and Figure 2-15 collaborate with the trends shown in Figure 13. Furthermore, it can be seen that research with CC is current between 2016 and 2021 in practically all countries. The research with SCC is also shown to be current in most countries. However, some countries no longer publish with relevance for about 8 to 10 years, as is the case of Italy and the United Kingdom.

2.5.8 Documents by area

The Figure 2-16 shows the distribution of published documents for CC and SCC for the areas of knowledge.



Conventional concrete (a)



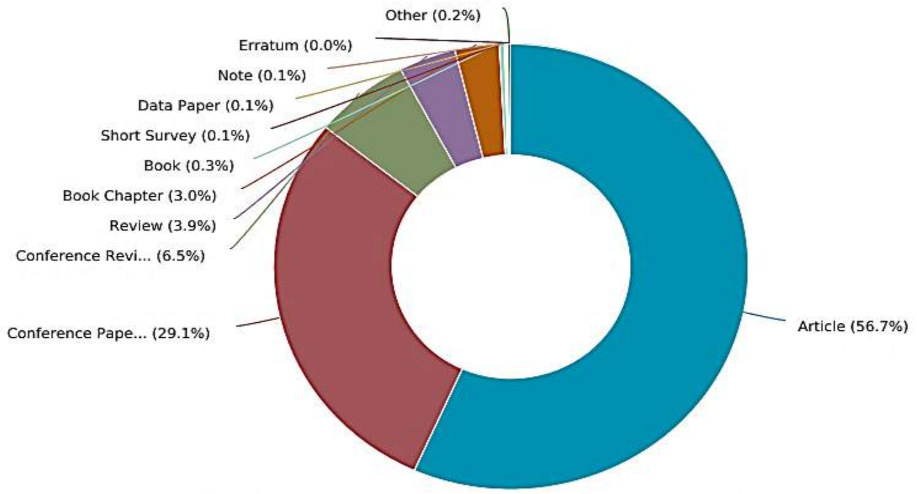
Self-compacting Concrete (b)

Figure 2-16: Documents by area for cement composite with recycled polymeric constituents on CC and SCC (AUTHOR, 2022).

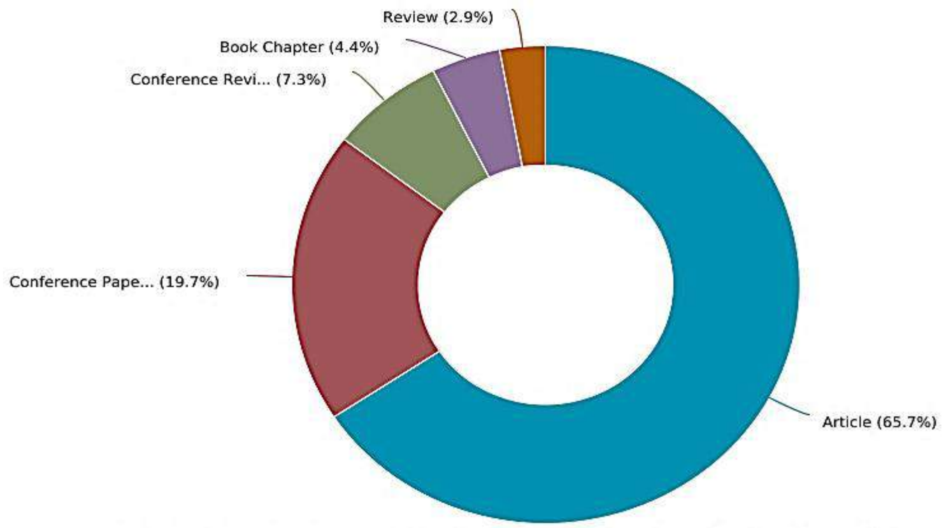
It is possible to identify that the areas of engineering, materials sciences, and environmental sciences were the most prominent in disseminating scientific material, both for CC and for SCC with waste. However, the areas of energy and physics appear in fourth and fifth place for CC. As for SCC, what changes is that the fourth and fifth places are areas of computer science and energy.

2.5.9 Documents by type

Figure 2-17 shows the distribution of published documents for CC and SCC for the different types of documents.



Conventional concrete (a)



Self-compacting Concrete (b)

Figure 2-17: Documents by type for cement composite with recycled polymeric constituents on CC and SCC (AUTHOR, 2022).

It is possible to identify that the main means of communication for SCC are articles (56.7%), conferences or congresses (29.1%), review conferences (6.5%), review articles (3.9%), and book chapters (3.0%). For SCC, the same classification order is repeated, for articles (65.7%), conferences or congresses (19.7%), review conferences (7.3%). However, in SCC, book chapters (4.4%) are more expressive than review articles (2.9%).

2.6 Conclusions

This study makes a bibliometric review on portland cement composite with recycled polymeric constituents of conventional concretes and self-compacting concretes with polymeric waste instead of natural aggregates. The following conclusions can be drawn:

In general, the methods adopted to carry out the research were satisfactory and made it possible to achieve the proposed objectives. The databases that generated the most results for CC were: Science Direct, Scopus, and Engineering Village, respectively. For SCC, they were: Science Direct and Engineering Village and Scopus, respectively. The keywords for both CC and SCC are linked to each other, so they are cited by authors from different areas. The main keywords for CC were mostly cited in the last five years, whereas in the keywords for SCC, there is a greater dispersion over time, with some words out-dated 8 to 10 years. The main keywords for CC are: concrete, compressive strength, mechanical properties, flexural strength, sustainability, fly ash, geopolymer, and crumb rubber. For SCC, they are: self-compacting concrete, mechanical properties, compressive strength, recycled aggregate.

The number of documents per year for both CC and SCC has grown exponentially since the 2000s and has continued to grow until the present moment. The main authors who publish in the area are segregated into individual research groups and have produced their work with greater volumes of studies in the last five years. The main journals are concentrated in European and Asian countries, and for CC, the production of the top 5 journals is over 20 documents per year, and for SCC, it is 1 to 3 per year.

The main institutions and main sponsors are from European and Asian countries. Highlighting China with a large number of institutions and a large volume of financial incentives for both CC and SCC. The countries that stand out the most for CC are India, USA, China, Malaysia, and Australia. For SCC, they are: India, China, Iraq, Iran, and Australia. Scientific production for both CC and SCC has some kind of link between countries and has been developed mainly in the last 8 to 5 years. The areas with the largest number of publications are: engineering, materials sciences, environmental and energy sciences, computer sciences. The types of documents most used as a means of publication for CC and SCC are: articles, conferences, and reviews.

With the data from this research, the perspective for the future of SCC and CC with polymeric residues is promising. Since the numbers of research in recent years are growing and there is involvement of several countries and researchers around the world. This also shows a proxy for world leaders (companies and politicians) with the environment and sustainable growth. This concern with the environment can be seen as a position in the predatory and unbalanced development of the last century.

The investigated theme is current and promising both for the development of concrete technology and for the environment with reuse of non-degradable material and replacement of natural aggregates. Since the approach of this study was carried out by the quantitative investigation of studies developed in the area of interest, thus, future studies are recommended to investigate which are the specific types of polymeric wastes most used, which are the main practical uses for concrete with polymeric wastes, and what are the financial and environmental impacts of the practice of reusing polymeric materials on a large scale in construction.

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3 DESIGN OF EXPERIMENTS TO OPTIMIZE USE OF POLYURETHANE RESIDUE IN CEMENT COMPOSITE

This study evaluates and optimizes the use of polyurethane powder in Portland cement mortars. Through a Design of Experiments Strategy (DOE), three manufacturing factors were considered and seven mechanical and physical responses were evaluated. The factors were modulus of fineness, percentages of replacement of polyurethane by fine aggregate and days of cure. The tested mortars obtained sufficient mechanical strength to be used in civil construction applications such as: coating and masonry laying. Mechanical strengths above 8 MPa for compression, 3.5 MPa for tensile and void index below 2.5% were obtained. Through optimization strategy applied in this study, an optimal configuration was obtained with a given arrangement that can support designers and engineers in projects that use polyurethane powder residue.

3.1 Introduction

Polyurethane is a polymer formed by the addition reaction between a cyanate that has the functional group [-N=C=O] (isocyanate), the group [-OH] (hydroxyl) both organic compounds [86]. The production of polyurethane on a world scale starts in 1940, its production increasing annually. Currently, the polyurethane polymer family is the most used and industrially produced. At the beginning of the last decade, global demand for polyurethane as raw material reached USD 43.2 billion with an average annual growth of 7.4% and is expected to reach approximately USD 66.4 billion by mid 2021 [87].

In general, experimental studies on the use of polymeric materials in cementitious materials began in 1930 with the addition of natural rubber in order to improve the workability, flexibility and tensile strength of cementitious compounds [88]. The first hypotheses raised for improvements in cementitious compounds with the use of polymers was to obtain an improvement in physical and mechanical properties, that is, resulting in a light, low-density material with good thermal insulation, workable and durable. It was assumed that concrete would acquire some of the qualities of polymers that are durable, ductile and light weight [89].

In the same way, Portland cement mortar is a cementitious product based on cement, sand, water and additives that can be used for sealing masonry, sealing and structural blocks [90]. New types of mortars are developed using polyurethane waste. The polyurethane-modified mortars have an advance in the reuse of waste, besides providing improvement in workability qualities, lower permeability, thermal and mechanical properties required range [91]. Mortar with polyurethane have a wide range of applications in construction their use can reduce the final costs and increase productivity, because the mortar gets better consistency [92].

Equally important, the reuse of polyurethane in mortar enables the reduction of industrial waste which minimizes environmental impact. The polyurethane is potentially more flexible and hydrophobic unlike most traditional lightweight aggregates. The mortars with polyurethane waste can be classified as light, because in the execution of the mixtures the polyurethane can be combined to substitute natural aggregates and obtain lower density and better workability [93].

Currently several studies are conducted with the objective of characterizing the behavior of lightweight mortars with polyurethane residue as in the studies of [94–96]. Mechanical strength such as tensile, modulus of elasticity, impact absorption, surface adhesion and hardness are parameters of critical importance in mortars. These issues were investigated by researchers [97–99] who identified ranges of polyurethane additions and substitutions in mortar, as well as use of nonionic sulfactants as an additive agent to promote adhesion. However, these mortar adhesion results have a variation that depends on the granulometry of the polyurethane used, type of mortar, environment used, selected raw material and other factors that involve the execution of the mortar according to the author. Considering this issue, the optimization of fresh and hardened characteristics of the mortars with polyurethane is also another much discussed theme [100]. Another important aspect and primary source of studies is the durability of mortars and concrete with polyurethane, this topic was studied by the following authors Muñoz R. (2018) [101] who identified that polyurethane can improve or not worsen the durable quality of the cementitious product, this will depend on the percentage added, granulometry and quality of the polyurethane. The polyurethane used in the study of Muñoz R. (2018) [101] in some mixtures reacted with alkalis in the pore solution, this reactivity of the compounds is proportional to the quality and purity of the polyurethane used. There is little evidence that polyurethane worsen the durable quality of mortars.

The properties and behavior of gypsum mortars designed with Polyurethane Foam Waste (PFW) are studied are investigated by [102]. The appropriate mechanical strengths were achieved, resulting in a new material that meets the requirements of the construction industry. The incorporation of ecological mortar with slag residue and polyurethane foam residue that was used to produce prefabricated panels [103]. The thermo physical and mechanical characterization of these panels shows a that the new material is suitable for use in civil construction. The chemical properties of the binders were modified with the use of non-ionic surfactants that improve the effect on clinker hydration [104]. This intervention provided mortars with densities 18.7% to 62.7% lower than conventional mortars without significantly decreasing mechanical strength.

The hydrophobic use in mortar with polyurethane residues is studied by [17]. The contact angle of lightweight mortars was determined (θ_w), as a function of time, before and after the frost resistance test. The surface free energy that characterizes the wettability and adhesion of mortars under normal conditions and after frost erosion was calculated by Neumann's method based on the obtained data. The best results illustrating the efficiency of hydrophobing were obtained for methyl silicone resin. Light structural eco-mortars made with polyurethane residues and non-ionic surfactants were studied by [94], obtaining mortars with low weight and better resistance. The fatigue durability of mortars with polyurethane foam residue was studied by [105]. The polyurethane foam mortars achieved approximately 5% fatigue strength. The investigation of gypsum composites with different lightweight materials, including polyurethane residues where it was possible to obtain more durable and 10% more impermeable gypsum composites, was carried out by [106].

In this work, the General Full Factorial design (GFFD) was used, both to plan the experiments and to analyze and optimize the responses. The main contributions of this manuscript are: i) collaboration with the development of cementitious composites using polymeric aggregates especially polyurethane; ii) simultaneous analysis of the influence of three influential factors of cementitious composites, granulometry, polyurethane percentage and curing time; iii) use of statistical strategy to evaluate the tested factors and determine the optimal or best possible accuracy configuration; iv) environmental contribution related to the incentive of using polymeric residues in cementitious composites, aiming at reusing this waste and obtaining mortars that have acceptable and/or better properties than those conventionally manufactured.

3.2 Methodology

3.2.1 Characterization of Material

The Portland cement used was the composite type with blast furnace slag type CPIIE32. The cement setting time starts at approximately 140 minutes and ends at 206 minutes after hydration. Local river sand and natural gravel were used as fine aggregate. The (potable) water used was from the public supply network of the local supply. In Figure 3-1 is shown the polyurethane in the two granulometries used and also the fine aggregate.

(a) (b) (c)

Figure 3-1: Fine aggregate used in the mortar: (a) River sand; (b) Polyurethane residue MF equal to 1.9; (c) Polyurethane residue MF equal to 3.1.

Na Table 3-1 is shown the characterization results performed for cement, sand and polyurethane. In Figure 3-2 is represented the granulometry curve for sand and polyurethane. For this study, the polyurethane was separated into two ranges of granulometry. The granulometries were classified by the fineness modulus. The fineness modules were 1.9 and 3.1, respectively. Aggregates with a fineness modulus equal to 1.9 are those passed through a 1.2 mm sieve. Aggregates with a fineness modulus equal to 3.1 are those retained in the 1.2 mm sieve. One can see the similarities between both and under these conditions the polyurethane works as fine aggregate in the mortar. To support the comparison, the limits of the usable zone and optimum zone determined by [107].

Table 3-1: Characterization of the materials used in the mortar

Ensaio	Cement	Sand	PU MF 1.9	PU MF 3.1
Density (g/cm³)	3.07	2.65	0.67	0.67
Unit mass (g/cm³)	1.03	1.46	0.22	0.19
Maximum Diameter (mm)	-	2.40	1.20	2.40
Modulus of fineness	-	2.5	1.9	3.1
Water absorption	-	2.41	0.30	0.25

PU MF 1.9: polyurethane with modulus of fineness 1.9;
PU MF 3.1: polyurethane with modulus of fineness 3.1.

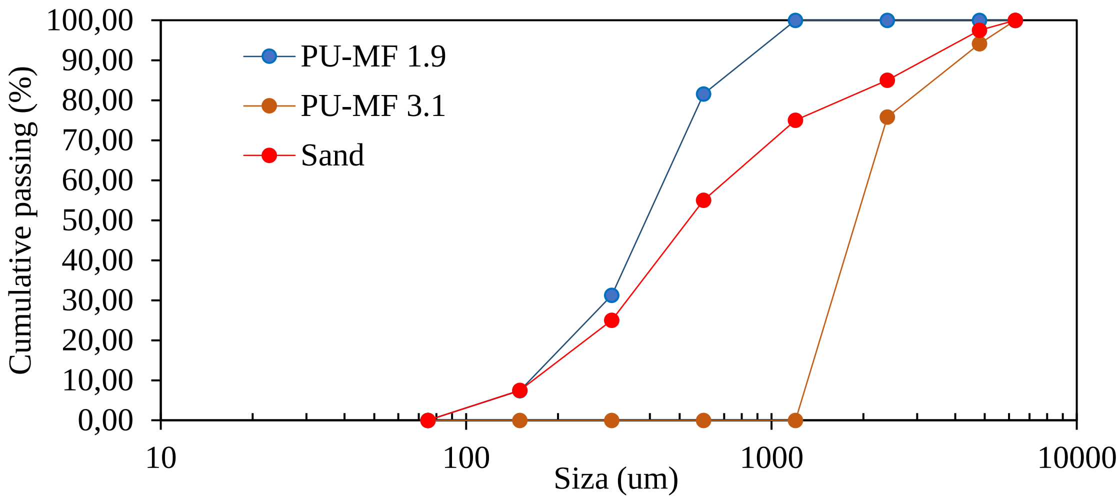

Figure 3-2:Granulometric curve sand and polyurethane.

In order to verify the composition and morphology of the polyurethane and compare it with the sand, as well as impurities that may be contained in this residue, a scanning electron microscopy (SEM) analysis was used, along with energy dispersive spectroscopy (EDS). In Figure 3-3 is shown the SEM analysis for the two aggregates, and

Table 3-2 the chemical elements found by EDS analysis of the polyurethane.

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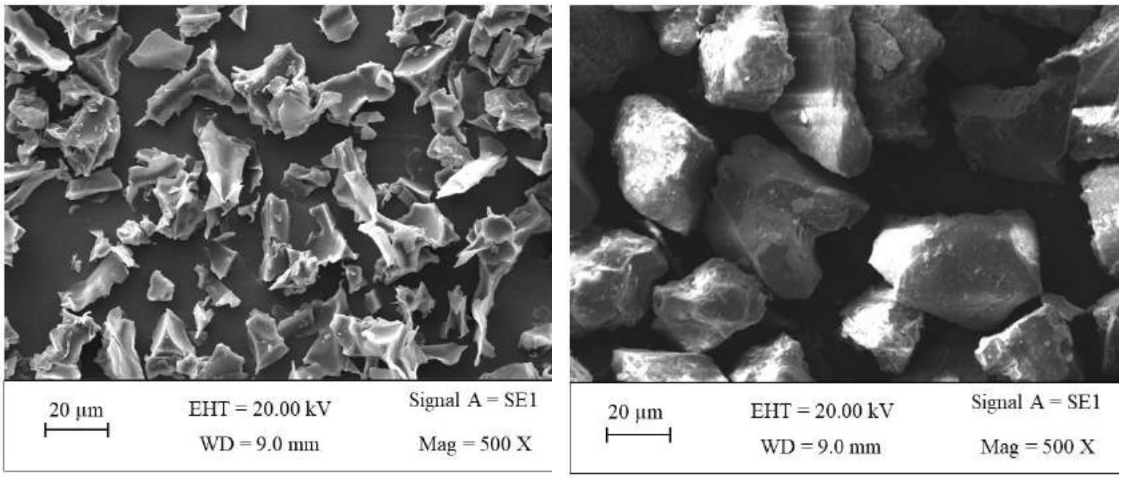

(a) (b)

Figure 3-3: Analysis of the microscopic form of PU (a) and sand (b).

Table 3-2: Analysis of PU composition

Elements	C	O	N	H	Na	Mg	Cl	Ca	Ti	Fe	Total
% Atomic composition	75.62	18.58	3.2	2.07	0.07	0.04	0.01	0.02	0.11	0.28	100.00

It is observed that the polyurethane has a pointed and lamellar morphology, and a predominant composition of carbon and oxygen, which is expected for a polymer. It is relevant to verify that there are no impurities detrimental to the hydration and crystallization reactions of Portland cement. That is, there is no significant presence of impurities such as metallic and semi-metallic elements. There is a large difference between the shape of the polyurethane particles and natural sand. The polyurethane is pointed and irregular in size in contrast to sand which is rounded and regular in dimension.

3.2.2 Mortar Manufacturing

The proportions of the materials were defined as being equivalent to a mix, by volume, of 1:1:6, when using natural sand, a trait widely used for laying and coating mortars [108]. The mortar developed used a 1:5 ratio, which means using one part of cement to five parts of sand, without using hydrated lime. In the authors' view, this would be a niche of activity in civil construction that would possibly benefit from mortars containing polyurethane as proposed in this study.

The polyurethane residue replaced the sand (fine aggregate) in volume. The variables used are summarized in Table 3-3. There are two different granulometries that result in different modulus fineness equal to 1.9 and 3.1. The lower modulus fineness of 1.9 was obtained by the particle size passing the 1.2 mm sieve, while the upper modulus fineness of 3.1 was obtained by the particle size retained on the 1.2 mm sieve. The percentages of polyurethane in relation to sand in mass correspond to 2.5%, 5% and 10%. And the curing time variables correspond to the tests for 7 days and 28 days of submerged curing. Differently from the properties in the hardened state in which it was measured at two different ages (i.e., 7 and 28 days of curing). The consumption and mass proportion of the materials is shown in Table 3-4.

Table 3-3: Experimental design for the mixtures

modulus fineness (MF)	Days of cure (D)	Polyurethane (PU)
1.9	7	0.00%
3.1	28	0.00%
1.9	7	2.50%
3.1	28	2.50%
1.9	7	5.00%
3.1	28	5.00%
1.9	7	10.00%
3.1	28	10.00%

Table 3-4: Proportion of materials in consumption per m³

Mixture	Cement (kg)	Sand (kg)	PU (kg)	Water (kg)
M0.0	333.1	1657.9	0.00	333.1
M2.5	333.1	1616.5	10.47	333.1
M5.0	333.1	1575.0	20.96	333.1
M10.0	333.1	1492.1	41.92	333.1

$\%V_{PU}$: Volume of PU replacing sand.

The mortars were produced using a 5 liter capacity benchtop mechanical mixer from Fortest and model ARG5 [109, 110]. The execution steps were as follows: (a) Mixing of cement and sand for 2 minutes at low speed; (b) Addition of polyurethane and mixing for 2 minutes at low speed; (c) Addition of water and mixing for 3 minutes at high speed (d) Execution of the verification of the fresh consistency, for this, 30 hits were executed at 30 second intervals and measurement of the spreading that should remain in the range of 260 mm (±20) value recommended for conventional mortar. The fresh mortar with polyurethane evaluation in the state rigorously follows the [110]. The samples were subjected to curing by immersion in water in $Ca(OH)_2$ solution for 7 days and 28 days of curing according to [111, 112].

3.2.3 Experimental Testing

Mortars were tested for consistency in the fresh state per the standard ABNT NBR 13276 (2016) [110]. Mortars with polyurethane were tested for mechanical strength and physical indexes. The tests that were performed are: uniaxial compressive strength ABNT NBR 7222 (2011) [113], flexural tensile strength ABNT NBR 7222 (2011) [113], dynamic modulus of elasticity ASTM C215 (2008) [114] and ASTM E1876 (2009) [115], density ABNT NBR 9778 (2009) [116], void index ABNT NBR 9778 (2009) [116] and capillary absorption ABNT NBR 9779 2012 [117]. Three replicates were used for each test.

In the hardened state the mortars were tested for axial compressive strength and tensile strength at 7 and 28 days of curing. Cylindrical specimens of 50 mm by 100 mm were molded in metal molds lubricated with mineral oil. A hydraulic loading machine from the brand Time testing machines model WAW-1000C, and load capacity 1000 Ton were used for the tests. The evaluation of the modulus of elasticity (E_d) was performed by means of the impulse excitation technique (TEI), at 7 and 28 days based on the studies of [118]. The calculation of E_d occurred through the method proposed by [119] that takes into account characteristics of natural aggregates, such as density and mineralogical compositions.

For the density and void ratio tests, dry mass was measured after a 24-hour period in an oven at 105°C (±5). Besides measuring the saturated mass and immersed mass, as recommended by the standard [116]. As for capillary absorption, the samples remained in an oven at a temperature of approximately 105°C (±5) for 24 hours, after which they were in contact with drinking water for 72 hours and saturated mass was measured. These procedures strictly followed the standard [117].

3.2.4 Design of Mortar Experiments

The design of the mixtures for mortar with polyurethane was planned based on requirements of the Brazilian standard ABNT NBR 13281 (2005) [120] in order to produce non-structural mortars for laying and coating masonry. After defining the test parameters and the range of use, the upper and lower limits of the mixture were defined. After this definition a General full factorial design (GFFD) was formulated and presents the factors at different levels.

The GFFD was chosen because there are two variables with two levels (modulus of fineness and curing time), and one variable with four levels (percentage of polyurethane). Thus, the GFFD was generated with three replicates, in Table 3-5 the control factors and the levels for each factor are shown. It is important to note that the experiments were performed in random order thus avoiding measurement biases.

Table 3-5: test variables and levels for the GFFD

Control factors	Symbol	Levels			
Modulus fineness (MF)	x_1	1.9	-	-	3.1
Polyurethane (PU)	x_2	0.0	2.5	5.0	10.0
Cure time (CT)	x_3	7	-	-	28

3.3 Results and discussion

The Table 3-6 shows in an ordered manner the test variables considered separated into continuous variable and categorical variable and all the results of the experiments. In the following sections the responses for each feature are evaluated using the Pareto Chart of Standardized Effects, Analysis of Variance (ANOVA) of the means, and analysis of the degree of linear regression (R^2). For the responses that are significant, linear regression equations that best fit the mathematical model will be generated. Finally, optimization conditions aimed at favorable maximization or minimization will be established using the statistically significant predictor variables. In Table 3-6 the positive and negative sign in percent indicates average increase or decrease with respect to the corresponding reference mixture.

The classification of mortars in terms of compressive strength according to ABNT NBR 13281 (2005) [121], varies from level P2 to P6 recommended by the standard for the use of mortar for laying and covering walls and ceilings. According to the standard ABNT NBR 13281 (2005) [121], this classification is indicated only for the purpose of documentation and quality control of the mortar. When the tensile strength in bending the mortars are classified in levels R1 to R2. When the density in bending the mortars are classified in levels D5 to D6. When the capillary coefficient, the mortars are classified in levels C1 to C4, with the vast majority classified as C2 and C3. At this point, it appears that the incorporation of polyurethane influenced the second classification of the mortars

[121]. In the next sections, it will be discussed, among other matters, whether the level of variation proves to be significant within the sample group tested.

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Table 3-6: Test results in GFFD proportions for mortar with PU

Variables				Responses													
#	MF	% PU	CT	y_0	Δy_0 [%]	y_1	Δy_1 [%]	y_2	Δy_2 [%]	y_3	Δy_3 [%]	y_4	Δy_4 [%]	y_5	Δy_5 [%]	y_6	Δy_6 [%]
1	1.9	0	7	259	-	5.11	-	1.17	-	8.26	-	2.60	-	14.34	-	3.12	-
2	1.9	2.5	7	271	4	4.90	-4.17	1.08	-8.04	2.44	-70.46	2.41	-7.30	13.89	-3.16	1.58	-49.24
3	1.9	5	7	245	-6	4.37	-14.42	0.96	-17.81	5.81	-29.66	2.25	-13.44	14.47	0.88	2.45	-21.56
4	1.9	10	7	218	-16	2.99	-41.55	0.86	-26.44	5.48	-33.66	2.24	-13.82	14.97	4.35	1.86	-40.37
5	1.9	0	28	259	-	8.39	-	1.96	-	19.74	-	2.59	-	10.54	-	4.27	-
6	1.9	2.5	28	271	4	7.32	-12.75	1.79	-8.34	17.92	-9.22	2.16	-16.84	9.64	-8.51	1.64	-61.48
7	1.9	5	28	245	-6	6.85	-18.36	1.45	-26.14	15.76	-20.15	2.23	-14.14	12.45	18.09	2.02	-52.66
8	1.9	10	28	218	-16	4.46	-46.88	1.27	-35.16	6.72	-65.93	2.13	-17.74	12.69	20.40	1.32	-69.14
9	3.1	0	7	259	-	5.11	-	1.17	-	8.26	-	2.60	-	14.34	-	3.12	-
10	3.1	2.5	7	286	10	3.59	-29.75	0.88	-24.50	2.60	-68.52	2.27	-12.93	14.27	-0.53	2.08	-33.21
11	3.1	5	7	255	-2	3.49	-31.77	1.12	-4.73	6.78	-17.96	2.22	-14.85	14.80	3.16	2.12	-32.04
12	3.1	10	7	243	-6	3.52	-31.18	1.03	-12.19	5.67	-31.32	2.14	-17.67	12.03	-16.13	1.73	-44.54
13	3.1	0	28	259	-	8.39	-	1.96	-	19.74	-	2.59	-	10.54	-	4.27	-
14	3.1	2.5	28	286	10	6.07	-27.61	1.49	-23.81	13.91	-29.54	2.21	-14.65	9.63	-8.63	1.64	-61.56
15	3.1	5	28	277	7	5.07	-39.57	1.27	-35.05	10.92	-44.67	2.26	-12.72	13.66	29.60	1.88	-56.02
16	3.1	10	28	243	-6	4.20	-49.98	0.91	-53.33	10.10	-48.83	2.18	-15.81	12.27	16.41	1.81	-57.58

y_0: spreading (mm), y_1: Compression Strength (MPa), y_2: Tensile strength (MPa), y_3: Modulus of elasticity (GPa),
y_4: Density (g/cm^3), y_5: Void index (%), y_6: Capillarity absorption (%).

The Figure 3-4 shows the moment of execution of several laboratory tests performed for the polyurethane mortar in the hardened state of this study.

(a)

(b)

(c)

(d)

Figure 3-4: Hardened state evaluation. (a) Verification of capillarity absorption; (b) Test of resistance to axial compression; (c) Tensile strength test; (d) Modulus of elasticity test.

3.3.1 Analysis of Variance (ANOVA)

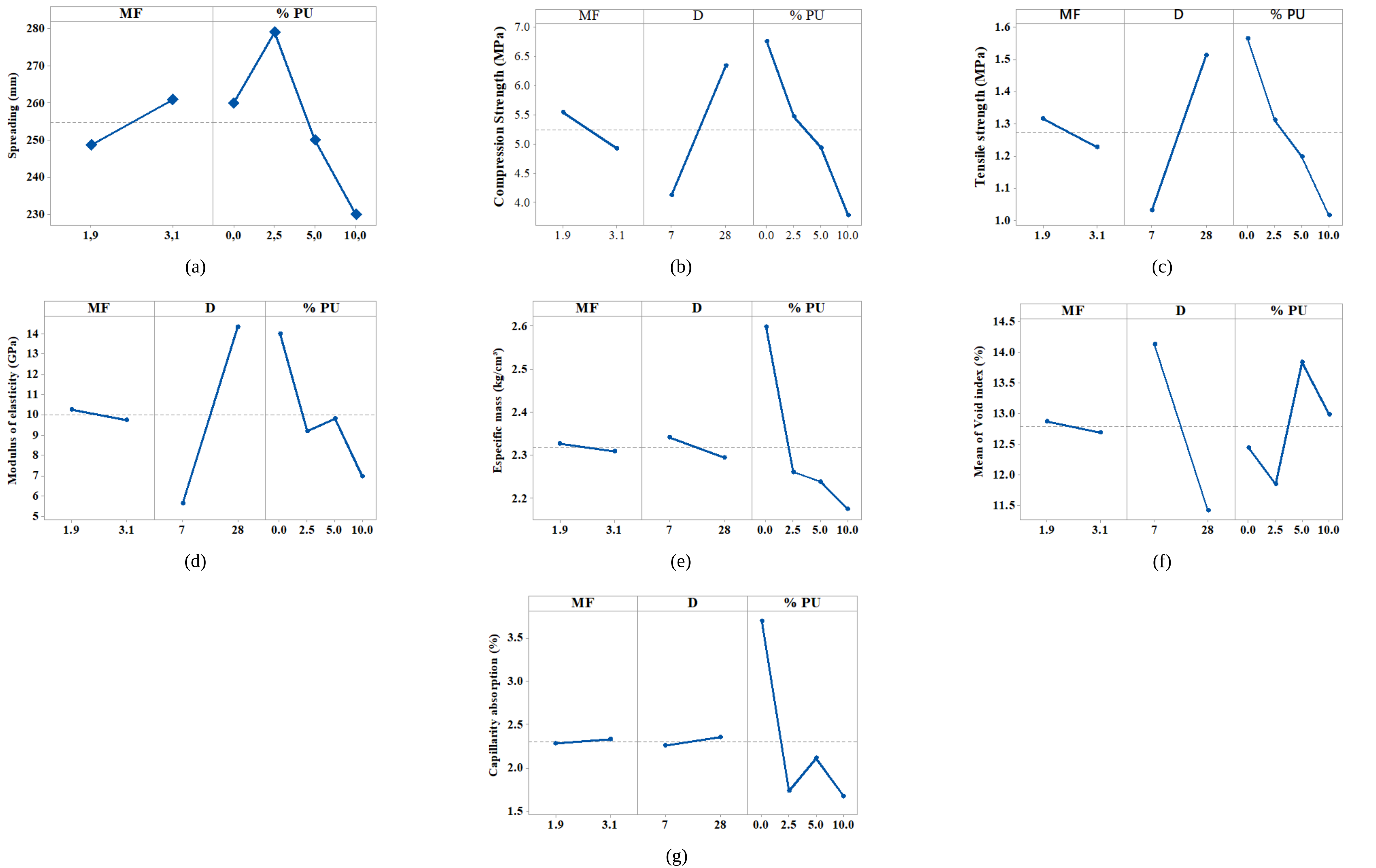

Figure 3-5:Main effects of the variables tested on the PU mortar. (a) Spreading of mortars; (b) Uniaxial compressive strength; (c) Flexural tensile strength; (d) Modulus of Elasticity; (e) Specific mass; (f) Void index; (g) Capillary absorption.

As shown in Figure 3-5(a), mortar spreading is proportional to fineness module, and for fineness module equal to 3.1 higher values of spreading are found. The percentage of polyurethane affects the spreading in a non-linear way, up to 2.5 there is an increase in the spreading, that is, the mortar loses viscosity. But for values greater than 2.5 there is a decrease in dispersion, i.e., increased cohesion. The flowability behavior of the mortar in question can be understood by thinking of the resulting fine aggregate as a mixture of aggregates and not as separate materials. That is, for each content of polyurethane replacing sand there is a mixture of a different nature. For the mixture of polyurethane aggregate plus natural sand at 2.5% polyurethane and 97.5% natural sand occurs the highest values of spreading. Funk and Dinger (1994) [122] have defined for ceramic materials the concept of Maximum Paste Thickness (MTP) and Interparticle Separation (IPS) which describes the largest average distance between particles in suspensions. Thus, the higher the MTP the easier mobility is achieved for a given water content. In the case of this study, the condition of continuity between the paste and aggregate phases is facilitated for fineness module contents equal to 3.1 and polyurethane content equal to 2.5%.

The polyurethane aggregate has low density and high surface area if compared to the natural sand, this can be verified in Table 3-1 and Figure 3-5, where the SEM image shows the irregular shape of the polyurethane and rounded shape of the sand. The Volumetric Surface Area (VSA) of the mixture is increased by the greater surface area of the polyurethane, but is decreased by its density. This balance probably has the best relationship at a ratio of 2.5% and fineness module equal to 3.1, since the VSA is inversely proportional to the mobility or flowability of the mortar or concrete mixture.

As shown in Figure 3-5 axial compressive strength is inversely proportional to fineness module and percentage of polyurethane, i.e., with increasing fineness module and percentage of polyurethane there is a decrease in compressive strength. However, the D variable is proportional to the axial compressive strength. The compressive strengths observed in the mortars demonstrate a behavior within the expected for the nature of the polymer incorporated into the mixture. Other authors such as Liu 2019 [123] and Arroyo 2019 [94];Vicario 2020 [124] also report great loss of mechanical strength with the incorporation of polymers in cementitious composites. The nature of polyurethane, generally is low strength, low weight and surface is irregular which imply in loss of mechanical strength, density and workability [125]. Another observed factor that contributes to the loss of compressive strength is the presence of the interfacial transition zone (ITZ). In conventional aggregates, the ITZ is the most fragile link of cementitious composites and the ITZ tends to be more fragile the larger the aggregate size. Large particles tend to attract more water around them, causing a point increase in the water-cement ratio [126]. This fact may help to understand why coarser particle sizes with fineness module equal to 3.1 achieve lower strength values (see Figure 3-5b).

The results for tensile strength are similar to those for compressive strength (see Figure 3-5c). In Table 3-6 it can be seen that the loss in compressive and tensile strength is greatest for fineness module equal to 3.1. However, for increasing amounts of polyurethane there are large losses in tensile strength. The tensile strength observed in the mixtures also shows similar behavior to that obtained by several authors [127–129]. The presence of the ITZ is also related to the poor tensile strength of the cementitious composites with both conventional and polymeric aggregate. For, in this region the greatest tendency of cracking concentration and higher porosity. From these regions the crack propagation occurs and this propagation is aggravated when there is no complete adherence of the aggregates and cement paste phases. In this case, the interfacial transition zone between cement paste and aggregate is compromised and makes the tensile strength of cementitious composites deficient [126].

As expected, (see Figure 3-5) the modulus of elasticity is proportional to the curing days and inversely proportional to the percentage of polyurethane. However, between substitutions of 2.5 to 5.0% a slight increase in the modulus of elasticity occurs. Modulus of elasticity is related to the interaction between the phases of cementitious composites. However, it is known that even the cement paste and natural aggregate phase has elastic behavior the cementitious composite as mortar and concrete has inelastic behavior. This is also related to the presence of the ITZ that compromises the deformation and does not allow the occurrence of flow thresholds [130, 131]. In the results of this study the incorporation of polyurethane gradually decreased the modulus of elasticity, a fact that is possibly linked to the nature of low stiffness of polyurethane, which is a flexible material compared to natural aggregate [132, 133].

In Figure 3-5(e) it is shown that the density is mainly affected by the percentage of polyurethane. The higher the substitution of polyurethane for sand, the lower is the density. This relationship occurs in a linear manner, a fact that does not occur for the fineness module and days of cure variables that have little effect on the density. Only by analyzing the percentage of polyurethane the variation in density reaches about 15% of alteration.

The voids index is inversely proportional to fineness module and days of cure, Figure 3-5(f). The influence of percentage of polyurethane is not linear with 2.5% being the lowest value. And from 2.5% an increase in the voids content occurs with a peak at 5.0% and a slight decrease to 10%.

With regard to density and void ratio, it is expected that lower density values will be found with the incorporation of lightweight aggregates. And this is one of the advantages of using polymeric aggregate [132]. The density is linked to the volume of voids, because the higher its value means a better packing between the particles in a given volume [134, 135]. As for the results of this study, there is a gradual loss of density with the incorporation of polyurethane, however, this decrease is not directly linked to the increase in the volume of voids.

As observed in Table 3-6, there are ranges of polyurethane incorporation that have a decrease in the volume of voids in the two granulometries analyzed. Probably the granulometric arrangement propitiated by the polyurethane grains accommodates and packs with more efficiency as the free space gel ratio increases. Add to this the fact that polyurethane residues have little resistance, and as the internal pressure increases, they give way and reduce in volume. According to Powers [136] the gel formed by C-S-H increases in volume with hydration time and up to a certain limit that will depend on the water-cement ratio and the content of aggregates per cement.

The capillary absorption in Figure 3-5(g) is indirectly influenced by percentage of polyurethane, with all samples with polyurethane having lower capillary absorption than the reference mixture. The other variables alone have little influence on the capillary absorption. Notably the absorption by capillarity is one of the most promising results found in this study. Loss of absorption is observed in almost all percentages of polyurethane incorporation, similar results were found by [137, 138]. Regardless of the granulometry used, the decrease occurs at higher levels for higher polyurethane percentages. Polyurethane has a hydrophobic nature, that is, it is repulsive and does not mix with water, and according to Table 3-1, the water absorption of polyurethane is approximately 87% lower than that of river sand. A fact that favors the use of polyurethane in mortar for placing masonry blocks, floors and coatings, because the resulting mortar will have greater durability by having little contact with water, besides the benefits of less proliferation of fungi and bacteria that occurs in walls with humidity [139].

3.3.2 Pareto of Standardized Effects

This section will discuss the results related to the standardized effects through the Pareto chart for the mortars with polyurethane. In Figure 3-6 it is shown all the results for the standardized effects according to the DOE analysis. The horizontal bars describe the effect of higher and lower relevance in the analyzed properties. The bars that exceed the reference line are statistically significant. For all results A stands for fineness modulus (MF), B percentage of polyurethane (%PU) and C Days of cure (D).

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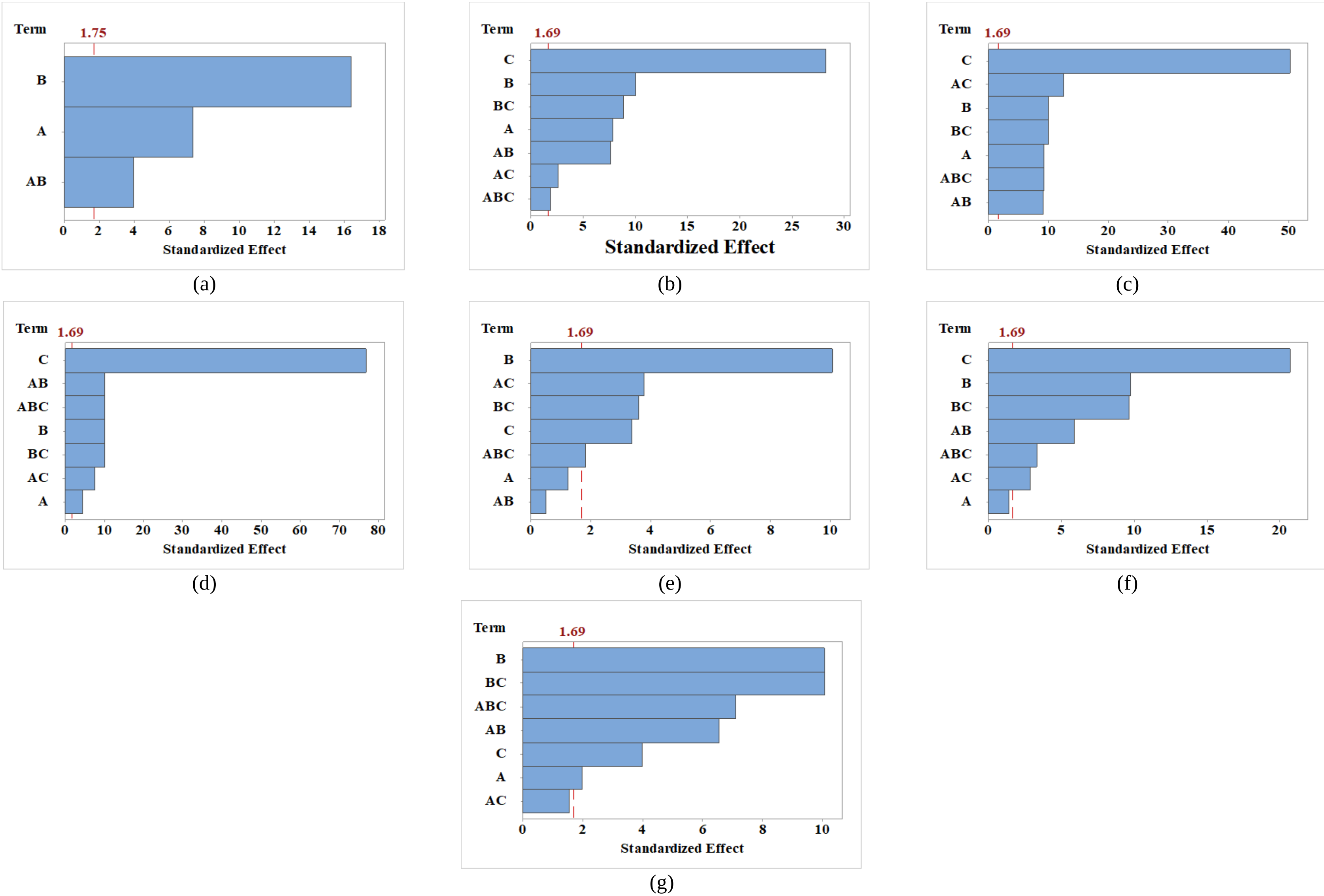

Figure 3-6: Standardized effects for mortar with PU. (a) Mortar spreading; (b) Compressive strength; (c) Tensile strength; (d) Modulus of Elasticity; (e) Density; (f) Void index; (g) Capillary absorption.

The mortar consistency by means of the spreading test is modified by both polyurethane incorporation and fineness module alteration, Figure 3-6(a). Being the percentage of polyurethane changes in a more accentuated way the mortar spreading. Also, the interaction between fineness module and percentage of polyurethane showed significant in modifying the spreading.

The compressive strength was altered by all the variables analyzed in this study, except the interaction of the three factors at the same time causes no significant effect, Figure 3-6(b). The days of cure is the factor that is most significant, followed by the percentage of polyurethane and interaction between days of cure and percentage of polyurethane. The fineness module even if to a lesser extent also significant in modifying the axial compressive strength.

The tensile strength is also strongly influenced by days of cure, Figure 3-6(c). All other factors analyzed are significant and exhibit equivalent effect, i.e., both percentage of polyurethane, fineness module and their interactions have the same strength in influencing the tensile strength.

The modulus of elasticity influenced by all variables analyzed, Figure 3-6(d). However, in different order of magnitude. The factor with the greatest impact is the days of cure, and the other factors are in equivalent order of influence.

In the density, Figure 3-6(e), the percentage of polyurethane is the most influential factor, with days of cure and interactions between days of cure, percentage of polyurethane and fineness module being significant to a lower extent. fineness module alone does not influence the density, but only when its influence with time is evaluated.

In the voids index, Figure 3-6(f), the variables that cause the greatest impact are days of cure and percentage of polyurethane, followed by the interaction between the two. Like the specific gravity, the voids volume is not affected by fineness module alone but only in interaction with days of cure and percentage of polyurethane.

The capillary absorption, Figure 3-6(g), is most affected by days of cure and percentage of polyurethane. The interaction between the variables days of cure, percentage of polyurethane and fineness module exerts great influence on the capillary absorption. However, fineness module alone and its interaction with days of cure does not show significant and alter the absorption.

The p-value values obtained were mostly less than 0.05, which means that we can reject the null hypothesis. There is a statistically significant difference between the tests performed, the variables and the combinations between them. The probability is almost zero that the test results will not be influenced if there is any change in the variables.

By analyzing 3-7 it is possible to perform a complete analysis of the behavior of the p-value for isolated variables (i.e., first order interactions) and their higher order interactions. Analyzing only the isolated variables it can be seen that in almost all cases the fineness module, days of cure and percentage of polyurethane obtained a significance lower than 0.05. That is, they were relevant in modifying the analyzed properties. Only for specific gravity and voids volume the fineness module is not significant, i.e., its presence does not cause significant changes.

The second order interactions, as well as first order interactions are mostly all significant. Only the interaction of variables fineness module and percentage of polyurethane in the density is not significant. The third order interactions are not significant in two cases. This is the case for compressive strength and specific gravity. However, for the other properties the third order interactions are significant.

Second and third order interactions can be understood as the dependence or correlation between two variables in a measured property. In this case the variables may have directly or inversely proportional correlations.

Table 3-7: p-value results for modal responses

Factor	Y0	Y1	Y2	Y3	Y4	Y5	Y6
MF	0.0000	0.0000	0.0000	0.0000	0.2080	0.1770	0.0540
% PU	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
CT	-	0.0000	0.0000	0.0000	0.0020	0.0000	0.0000
2-Way Interactions							
MF × CT	-	0.0170	0.0000	0.0000	0.0000	0.0080	0.1290
MF × % PU	0.0010	0.0000	0.0000	0.0000	0.5960	0.0000	0.0000
CT × % PU	-	0.0000	0.0000	0.0000	0.0010	0.0000	0.0000
3-Way Interactions							
MF × CT × % PU	-	0.0810	0.0000	0.0000	0.0660	0.0020	0.0000

Legend: MF: fineness modulus; PU: percentage of polyurethane; CD: cure days.

In Table 3-8 is shown the values of R^2 that defines the degree of fit of the mathematical model that is most accurate for values close to 100% [140]. The responses for almost all the properties analyzed were high (i.e. above 90%). This means that the mathematical model taking into account the predictor variables fits very well to describe the analyzed properties.

Table 3-8: Adjustment of the linear model by regression analysis

Responses	R^2	R^2 adj	R^2 pred
y0	97.13%	95.88%	93.54%
y1	98.26%	97.28%	95.55%
y2	99.58%	99.34%	98.92%
y3	99.69%	99.51%	99.21%
y4	95.56%	93.04%	88.62%
y5	96.14%	93.95%	90.11%

The following is a set of mathematical equations that translate the ability to generate responses through mathematical equations. In this model, the manufacturing variables (x_1, x_2, x_3) and their interactions (x_1x_2, x_1x_3, x_2x_3, $x_1x_2x_3$) are presented. The equations y_0 to y_6 represent the result of the mathematical model for each tested characteristic. The variables x_1, x_2 and x_3 represent MF, %PU and MF respectively. Thus, the equations represent the best linear configuration according to the factorial design adopted.

The following is a set of mathematical equations that translate the ability to generate responses through mathematical equations. In this model, the manufacturing variables (x_1, x_2, x_3) and their interactions (x_1x_2, x_1x_3, x_2x_3, $x_1x_2x_3$) are presented. The equations y_0 to y_6 represent the result of the mathematical model for each trait tested. The variables x_1, x_2 and x_3 represent MF, %PU and MF respectively. Thus, the equations Eq 3-1 to Eq 3-7 represent the best linear configuration according to the factorial design adopted.

$$y_0 = 281.40 + 4.22x_1 - 7.71x_2 + 0.36x_1x_2 \quad \text{Eq 3-1}$$

$$y_1 = 5.525 - 0.582x_1 - 0.278x_2 + 0.139x_3 + 0.063x_1x_2 - 0.009x_2x_3 \quad \text{Eq 3-2}$$

$$y_2 = 567.8 - 107.2x_1 - 54.72x_2 + 38.31x_3 + 22.09x_1x_2 + 0.435x_1x_3 - 0.395x_2x_3 - 0.873x_1x_2x_3 \quad \text{Eq 3-3}$$

$$y_3 = -6.727 + 0.785x_1 + 1.231x_2 + 1.152x_3 - 0.108x_1x_2 - 0.105x_1x_3 - 0.118x_2x_3 + 0.016x_1x_2x_3 \quad \text{Eq 3-4}$$

$$y_4 = 2.655 - 0.061x_1 - 0.025x_2 - 0.019x_3 + 0.003x_1x_3 + 0.001x_2x_3 \quad \text{Eq 3-5}$$

$$y_5 = 14.005 + 0.645x_1 + 0.399x_2 - 0.195x_3 - 0.190x_1x_2 - 0.022x_1x_3 + 0.001x_2x_3 + 0.006x_1x_2x_3 \quad \text{Eq 3-6}$$

$$y_6 = 0.802 + 0.334x_1 + 0.144x_2 + 0.038x_3 - 0.045x_1x_2 - 0.014x_1x_3 - 0.008x_2x_3 + 0.002x_1x_2x_3 \quad \text{Eq 3-7}$$

3.3.3 Optimal Setup

In Table 3-9 is presented the optimization values for the properties measured in the mortars with PU. The optimizations were performed to find the desirable properties for mortar that can be used in masonry block and wall covering, as this would be the most likely application of the PU mortars in this study. The mortars for laying and coating need to be easy to apply to achieve the best possible adhesion to the substrate [141, 142]. In general, the compressive strength cannot be too high; some studies show an indirect relationship between compressive strength and mortar adherence to the substrate [143, 144]. But the tensile strength should be as high as possible to withstand the bending imposed by the deformation that buildings usually suffer throughout their service life [145, 146].

The modulus of elasticity property also known as modulus of rigidity describes the deformation that a material will have for a given stress. It is preferable that mortars are able to deform as much as possible before entering the plastic state and cracking. It is known that materials with low modulus of elasticity can deform more before cracking [147]. Finally, it is desirable that the properties of Density, void volume and absorption are minimized, thus resulting among other benefits, lower weight per unit area, less contact with aggressive agents from the atmosphere and contact with water [148].

The requirements listed in order from 1 to 7 represent the optimization of properties independently. Item 8 represents the optimization of properties simultaneously for all properties.

Table 3-9: Optimized configurations for the analyzed mortar properties

Order	Objective	Response	Optimal Design			Predicted Response
			MF	PU	CT	
1	Maximization	Spreading	3.1	2.50	-	286.67 mm
2	Target	Compressive strength	1.9	10.00	28	2.00 MPa
3	Maximization	Tensile strength	1.9	2.50	28	1.29 MPa
4	Minimization	Module of elasticity	1.9	2.50	28	2.44 GPa
5	Minimization	Density	1.9	10.00	28	2.13 g/cm³
6	Minimization	Void ratio	3.1	2.50	28	9.63
7	Minimization	Capillary absorption	1.9	10.00	28	1.32
8	Maximization	Spreading	1.9	10.0	28	286.67 mm
	Target	Compressive strength				2.00 MPa
	Maximization	Tensile strength				0.99 MPa
	Minimization	Module of elasticity				13.91 GPa
	Minimization	Density				2.21 g/cm³
	Minimization	Void ratio				9.63
	Minimization	Capillary absorption				1.64

From Table 3-9 it can be concluded that the optimization is more efficient when the values of the properties are optimized individually. In general, there is a decrease in the mechanical properties with increasing percentage of PU incorporation, similar results were found by [7].

In item 8, where the simultaneous optimization of properties was performed, there is a greater use of the 10% PU. This in general is beneficial to the environment, because there is a greater use of polyurethane waste. It should be understood that this simultaneous optimization is not as efficient as the individual optimization for each property. However, it is a good starting point for dosing mortars that use PU and want to make the best use of all the properties presented here together.

Due mainly to the type of residue that is a polymer of low mechanical strength, irregular shape and low specific mass it is to be expected that a decrease in the mechanical strength of the mortar will occur. Also Kujawa w. (2021) [89] points out that the loss of strength in cementitious composites is closely linked to the weak interfacial zone between cementitious composite matrices and polymeric materials.

The values of physical properties such as specific mass, void index and water absorption generally follow to a decrease with increasing PU content, as also observed in the studies of [17, 149–151]. According to Wu H. (2022) [87], mortar and concrete by themselves are heterogeneous materials, most composite materials follow the properties of their constituents, given the degree of proportion used it is natural that a decrease in specific mass is obtained. However, the decrease in absorption and void ratio is not so simple to analyze, because it depends on a number of factors such as the degree of interaction of the components, particle packing, and the characteristics of each phase of the mixture [152]. In Figure 3-7 and Figure 3-8 are the surfaces generated by the regression equations obtained through factorial analysis. These surfaces are drawn in the analysis range for each predictor variable and reflect the good adjustment obtained in the linear model regression.

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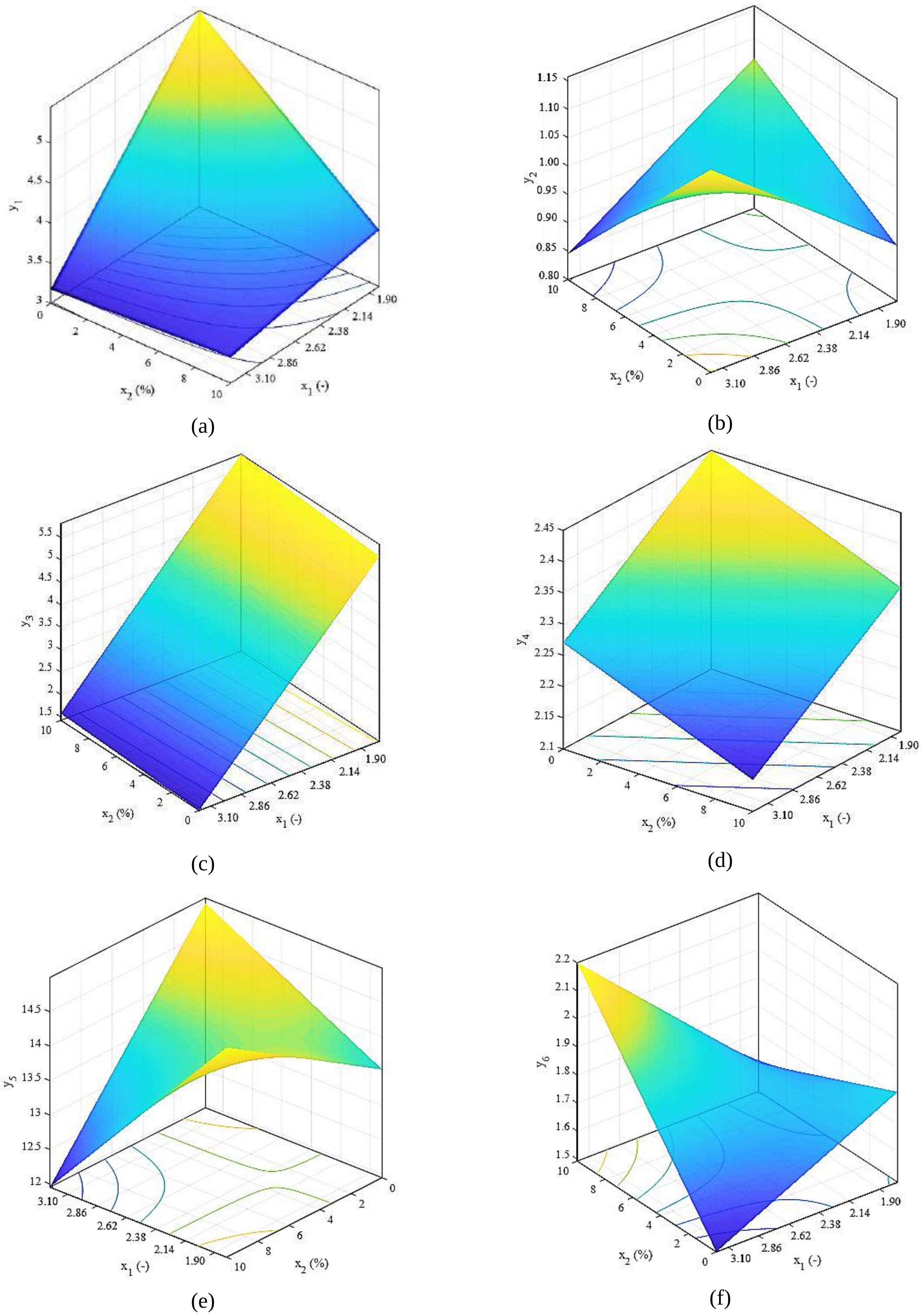

Figure 3-7: Full factorial regression results for curing time of 7 days. (a) Compressive strength; (b) Tensile strength; (c) Modulus of Elasticity; (d) Density; (e) Void index; (f) Capillary absorption. Legend: y1[MPa]; y2[MPa]; y3[MPa]; y4[GPa]; y5[%]; y6[%].

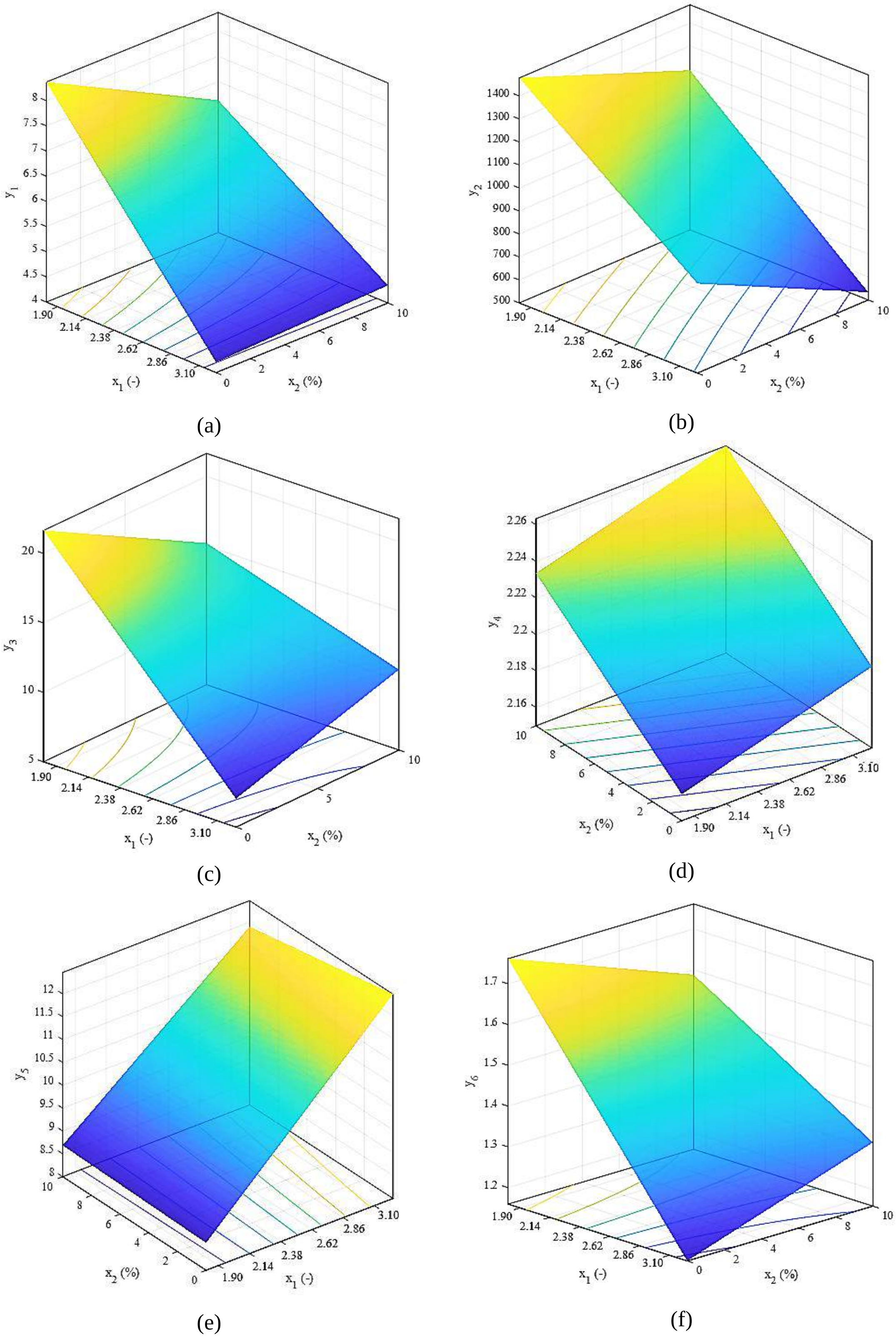

Figure 3-8: Full factorial regression results for curing time of 28 days. (a) Compressive strength; (b) Tensile strength; (c) Modulus of Elasticity; (d) Density; (e) Void index; (f) Capillary absorption. Legend: y1[MPa]; y2[MPa]; y3[MPa]; y4[GPa]; y5[%]; y6[%].

Through a theoretical and practical approach to the use of polyurethane waste in cementitious mortars for use in construction. This study sought to show that it is possible to parameterize known indexes of the characteristics of aggregate properties, such as fineness modulus and hydration time to accurately correlate some properties of mortars in the fresh state, such as spreading and mechanical strength, as well as physical indexes such as water absorption. Based on the ABNT NBR 13281 (2005) [121], it is possible to qualify the use of mortars with polyurethane in this study as suitable for use in laying and covering walls and ceilings in civil construction.

3.4 Conclusion

In this study an experimental program was developed to evaluate the behavior of mortars with polyurethane as a replacement for fine aggregate. The aim was to optimize the use of polyurethane residue by varying the fineness modulus (granulometry), curing time and percentage of substitution using the DOE statistical technique. The following conclusions can be drawn from this study.

The mortars obtained higher values of spreading between 2.5% of polyurethane and fineness module equal to 3.1, reaching an increase of 10%. The uniaxial compressive strength, flexural tensile strength and dynamic modulus of elasticity were negatively affected by polyurethane incorporation with decreases ranging from 4% to 72%. However, the loss of resistance was lower when using a particle size fineness module equal to 1.9 and for 2.5% contents of polyurethane replacing sand.

The density of the mortars decreased up to 18%. The variation of the voids index did not occur in a linear pattern. For 2.5% incorporations there is a decrease of up to -9% of the voids index, but from 5% of polyurethane there is an increase in the voids index of up to 30%. Water absorption decreases in all polyurethane incorporations, reaching a decrease of up to 60%.

The model obtained has the great advantage of being a guide for designers and construction professionals who will not need to perform a whole set of laboratory experiments with mortars to obtain the appropriate proportions for each project. The mathematical model obtained is capable of generating answers with remarkable accuracy, as seen by the excellent fit obtained. Moreover, all the variable analyses performed exhibited sensitivity of the responses as a function of the predictor variables adopted. In summary, it can be said that the models evaluated in this study have an excellent linear correlation for variable decision and response analysis.

4 RSM-BASED MODELING AND OPTIMIZATION OF CEMENTITIOUS COMPOSITES WITH POLYURETHANE POWDER WASTE AND FOUNDRY EXHAUST SAND

In order to generate sustainable mortars, the current study will look at the impacts of adding two wastes into Portland cement mortar: polyurethane powder waste (PU) and foundry exhaust sand (FES). The tests were carried out utilizing an RSM configuration with four different manufacturing variables: sand/cement ratio, coarse sand content, FES, and PU replacing natural sand. Compressive strength, modulus of elasticity, void index, and water absorption all showed substantial fitting values in the data. Furthermore, the experimental findings show that the adopted model is capable of accurately predicting the outcomes, and an optimized mortar combination is discovered and presented.

4.1 Introduction

The globe is witnessing a new social revolution that aims to promote awareness about climate issues and the hazards linked with climate change, which might result in drastic lifestyle adjustments [1]. A new social model in many countries is being implemented that seeks alternatives to traditional industry, avoiding or minimizing environmental impacts that contaminate and pollute, thus mitigating the effect of global warming and overexploitation of natural resources [2]. Efforts must be made to avoid the collapse of the progress of today's civilization. Efforts must be made to reduce our dependence on traditional raw materials, thus looking for valid alternatives through recycling and recovery of industrial waste seems to be the most viable way forward [3, 4].

The two wastes explored in this study are PU and FES, which are of different physical and chemical natures but which impact the environment in an equivalently negative way [5]. The PU originates from the polymer family, so it is very versatile and can come in many shapes and sizes and be used on a large industrial scale [6]. This product has a low production cost and high durability and is widely used in the construction sector and in the household appliance trade in general [4, 7]. On the other hand, foundry sand is a by-product of metallurgical production. The so-called foundry sand, or green sand, is incorporated into molds that can be reused several times during the production of metal parts [8, 9]. During part manufacturing, factory exhaust fans capture high surface area sand residue that comes loose in the molds. FES is the airborne residue that comes loose from molds and can be captured by exhaust fans. Thus, FES is generally finer than conventional exhaust sand [10]. And because it is thinner, its disposal in the environment is difficult due to the numerous possibilities of contamination for the local fauna and flora [11].

Overall, the average world production of plastics over the last decade has been 300 million tons per year. However, only 14% of polymeric waste is collected for recycling. Of this, only 9% is actually recycled and returned to the market [15]. Thus, it is necessary to develop studies that aim to adapt the use of PU waste in civil construction to its adaptation, because this is an area that consumes a large amount of non-renewable raw materials. There are several current studies on the use of PU in civil construction. [16] It uses PU to perform some of the ballast work for highways and finds that the use of PU results in ballast with better freeze-thaw strengths, fatigue resistance, and less residual deformation. Briones L. (2020) [4] execution of ecological mortars composed of PU and Portland cement for the production of blocks and panels for hospital construction. Simulations of the energy behavior of the hypothetical building were generated, proving that the new material can help with the thermal comfort of buildings. Santamaría V. (2020) [7] Studying plaster mortar with PU results in composites that are less permeable and suitable for application in residential construction walls. Barnat H. (2018) [17] study mortars with PU and hydrophobic agents, obtaining adequate results for contact angle and freeze and thaw resistance. Also, the surface free energy, surface wettability, and adhesion under normal use conditions were determined by Neumann's method.

The FS has shown great potential for use in civil engineering. Vilenius (2019) [18] investigates the feasibility of applying FS to concrete for highway construction and proves its viability as a stabilization layer, serving as a load-bearing layer of a sidewalk structure that is suitable for unconfined compressive strength. Yaghoubi (2020) [153] produced an independent test series and concluded that FS in proper mix proportions and with well graded aggregates can work as load-bearing structures. FS is widely applied in the production of low-strength materials or as a substitute for fine and coarse aggregate for cementitious composites [20–22]. However, its application contains negative points that need to be evaluated. High levels of substitution of FES fine aggregate for coarse aggregate (natural sand) is reported to be detrimental to the mechanical strength and freeze-thaw resistance of cementitious composite mixtures [23].

The manufacture of cementitious products such as mortars and concrete can benefit from technologies that determine the optimization of the dosage process through a statistical approach [24]. Several successful approaches have been used for optimal modeling in concrete technology, such as artificial neural networks [25, 26], genetic algorithm [27, 28], neuro-fuzzy inference system (ANFIS) [29].

Another interesting approach is the use of Design of Experiment (DOE), which is a statistical method used to plan and analyze the results of experiments. The use of DOE can reduce the number of experiments, help evaluate the relationships among variables, provide a mathematical model, and obtain optimal experimental results [30]. Also, using the mathematical model, the results of a different set of parameters can be predicted [31].

Within the DOE approach, there is Response Surface Methodology (RSM), which is a sub-item of DOE widely used for optimization of industrial procedures [32]. Considering the rules of mathematics and statistics, RSM not only reveals the effects of different variables on the desired parameters but also portrays the effects of the interaction between the variables on 3D surfaces. The accuracy of this method for evaluating mechanical, physical, and durability properties of cementitious products such as concrete and mortars is recognized to be adequate and reliable [33].

The overall objective of this study is to evaluate the various mechanical and physical properties of MPUFS simultaneously using RSM. Thus, PU and FES were considered as partial substitutes for natural sand, and modifications of these variables were investigated. Other variables investigated were sand to cement ratio, S/C, and SC coarse sand content in the variation of properties. In this way, an ecological mortar is sought to be produced with relatively low cement consumption and the reuse of natural resources. The optimization process was based on four hypothetical scenarios in arrangements that were aimed at the maximum benefit of the mechanical and physical properties and the maximum percentage of use of PU and FES waste. Thus, this study presents a current approach using two already known residues in isolation, but in a novel way with a joint approach of PU and FES.

4.2 Experimental procedure

4.2.1 Materials

The materials used in this study were: cement type CP II-E-32 [154], hydrated lime ABNT NBR 7175 (2003) [155] as binders, natural sand (NS), foundry exhaust sand

(FES), polyurethane powder (PU) as aggregate. The chemical composition and physical properties are in Table 4-1 and Table 4-2, respectively. The Figure 4-1 represents the particle distribution of the binder (cement and lime), obtained by the laser ray diffraction method and aggregates (NS, FES, PU) obtained by sieving on the normal sieve series [156, 157]. Drinking water was used for molding and curing the samples. Furthermore, according to [158], 1.5% of water reducing admixture, 3rd generation superplasticizer based on modified synthetic carboxylated polymers with specific weight of 1.24 kg/lit) was used to increase the consistency of the MPUFS with the lowest possible (w/b) ratio.

Table 4-1: Chemical composition and elements of cement and aggregates

Compounds	Cement %	Lime %	FES %	Elements	PU %	NS %
SiO_2	18.63	1.88	81.08	C	40.56	12.93
Al_2O_3	2.31	0.26	1.22	H	34.05	32.75
Fe_2O_3	5.00	0.22	15.97	O	14.55	3.70
CaO	64.21	49.35	0.20	N	9.33	21.71
MgO	6.20	0.00	0.38	Al	0.05	0.00
SO_3	4.74	0.00	0.00	Si	0.04	0.00
Na_2O	0.85	0.00	0.40	Fe	0.03	0.16
K_2O	1.55	0.06	0.00	Zn	0.03	0.19
MnO	1.12	0.00	0.12	Na	0.01	1.38
TiO	1.23	0.00	0.11			0.16
MgO	0.00	26.45	0.00			
C_3S	71.19					1.17
C_2S	12.37					
C_3A	7.71					
C_4AF	14.03					

Table 4-2: Physical properties of fine and coarse aggregates

Properties	Cement	Lime	NS	PU	FES
Density (kg/m^3)	3140	1600	2650	660	2700
Specific surface (m^2/kg)	330		5	20	29.4
Initial setting time (min)	120				
Final setting time (min)	500				
Water absortion			0.95	0.05	0.75
Unit Weight (kg/m^3)	1440	1120	1350	220	1450

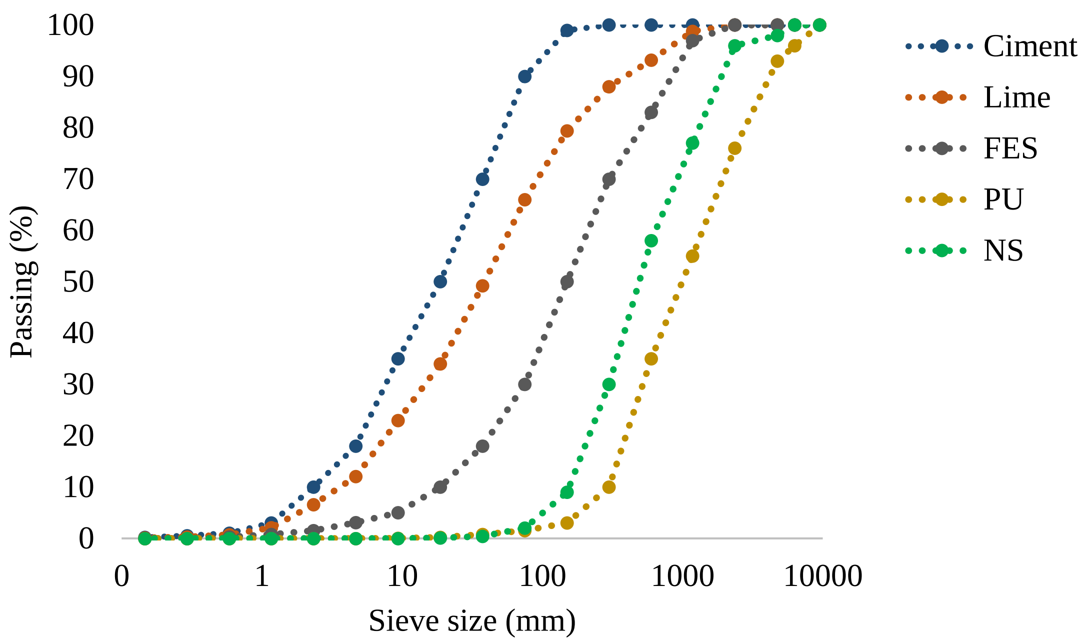

Figure 4-1: Particle size distribution of cement, SF and GGBFS

In this study FES and PU were used as fine aggregate and substituted the NS in mass in different percentages. The PU used comes from the recycling of household appliances, more specifically from domestic refrigerators; it was obtained through a donation from the company Fox Industry®. According to surveys conducted by Fox Industry®, a company specialized in recycling this type of appliance, refrigerators and freezers contribute with 200 thousand tons of discarded residues per year in Brazil. In Figure 4-2 is shown the deposit of refrigerators and the PU resulting from recycling. According to Fox Industry® the recycling process used by the company consists first in filtering the refrigeration gases Chlorofluorocarbon (CFC) that contribute to the depletion of the ozone layer, and persistent organic pollutants such as mercury present in the refrigerator compressor. These gases are treated by a chemical process that transforms the gas into an acid solution, so it can be used later by the chemical industry. What is left over from the device is ground up and the remains of plastic, iron, aluminum, and any other materials that can be reused are separated and forwarded to recycling companies and cooperatives [159].

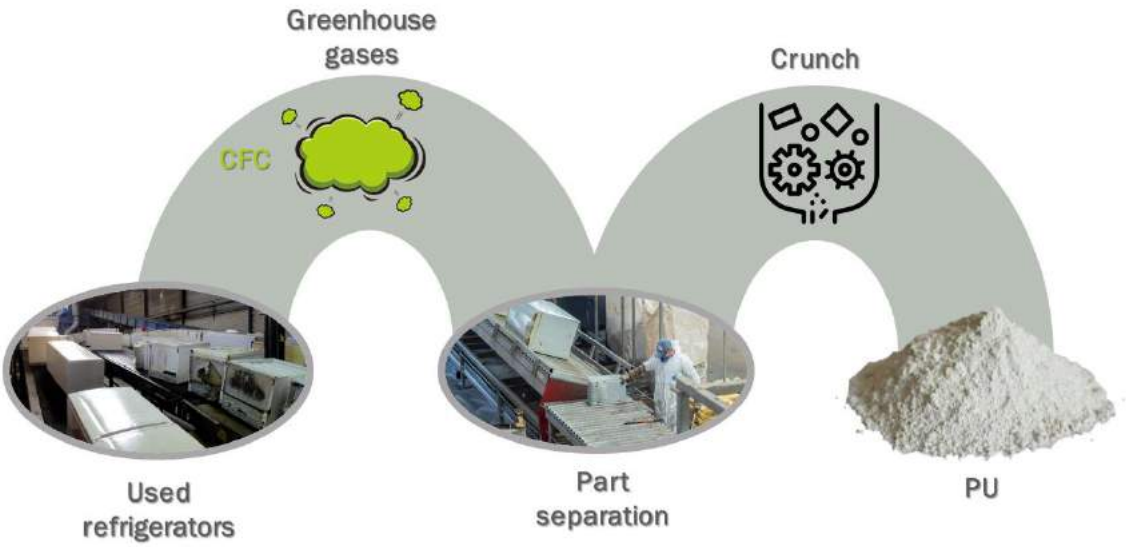

Figure 4-2: Phases performed by Fox Industries to obtain PU.

The foundry waste industry generates numerous by-products, ranging from used or residual foundry sand, slag, ash, refractories, coagulant, dust from the exhaust system (filters), scrap, vapors, and residual kiln liquids [10, 160]. In this study, the FES was obtained through donation from the company Mahle Metal Leve AS®, Itajubá. The FES is a type of foundry sand waste that is generated by mixing sand, bentonite and charcoal in the manufacturing process of green sand molds for casting metallic parts. During the demolding of the parts by means of vibrating transport, the very fine sand particles that remain in suspension are filtered by baghouse filters Martins (2021) [161], the process of casting and obtaining FES is briefly illustrated in Figure 4-3. It is necessary to remove the FES from the casting process, because due to its powdery size it ends up impairing the permeability of the molds and hindering the escape of gases from the process. With this, harmful bubbles and voids can be formed in the castings [162].

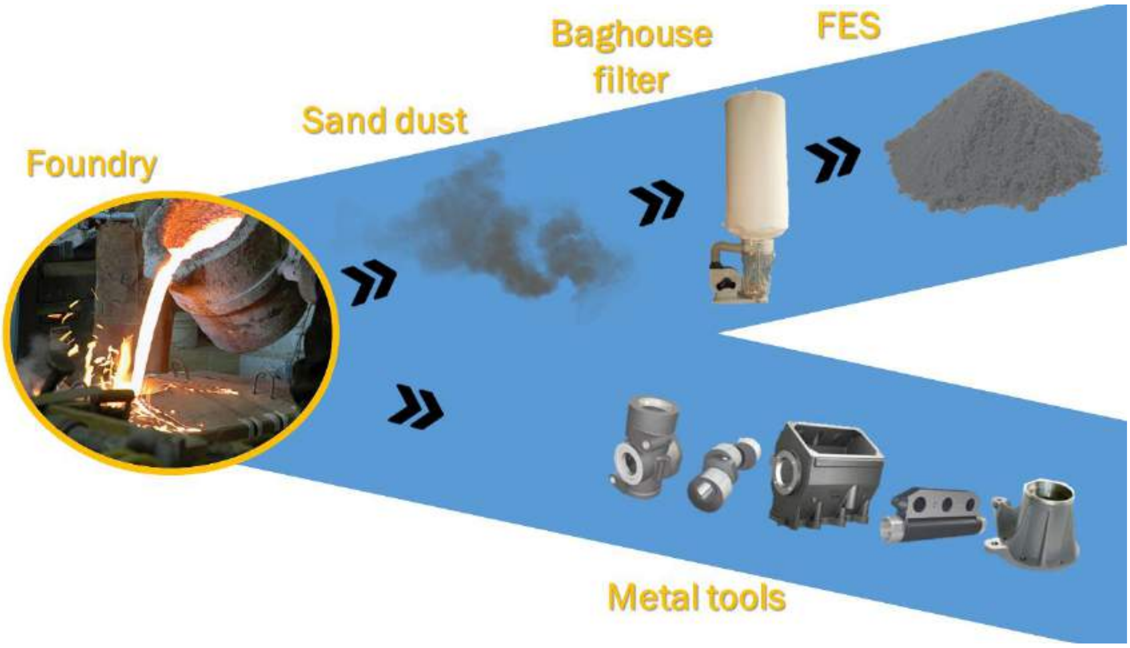

Figure 4-3: Process of obtaining FES by the company Mahle Metal Leve AS®.

4.2.2 RSM theoretical model

The mathematical RSM model is widely used when there are one or more variables influencing one or more responses. Thus, RSM considers the interaction between independent variables (input parameters) and one or more responses (output parameters). In this way, it is able to provide an accurate estimation of the model with a small number of experimental data [163].

In this study a Box-Behnken Design (BBD) was developed for each response, which is an experimental RSM model that is capable of predicting a second-order (quadratic) model. The procedures followed in this study are schematically shown in Figure 4-4. The variables and their ranges are given in Table 4-6. Thus, there are 3 levels with 4 control factors S/C, CS, FES, and PU. In the RSM BB setup, alpha equal to one ($\alpha = 1$) was used so that the axial points are on the faces of the experimental cube. This type of arrangement maintains the orthogonality of the experimental points. However, RSM-BB is limited in not having rotationality of the points that is achievable in RSM-CCD (Central composite design), but this fact does not diminish the validity and accuracy of the RSM-BB methodology [164].

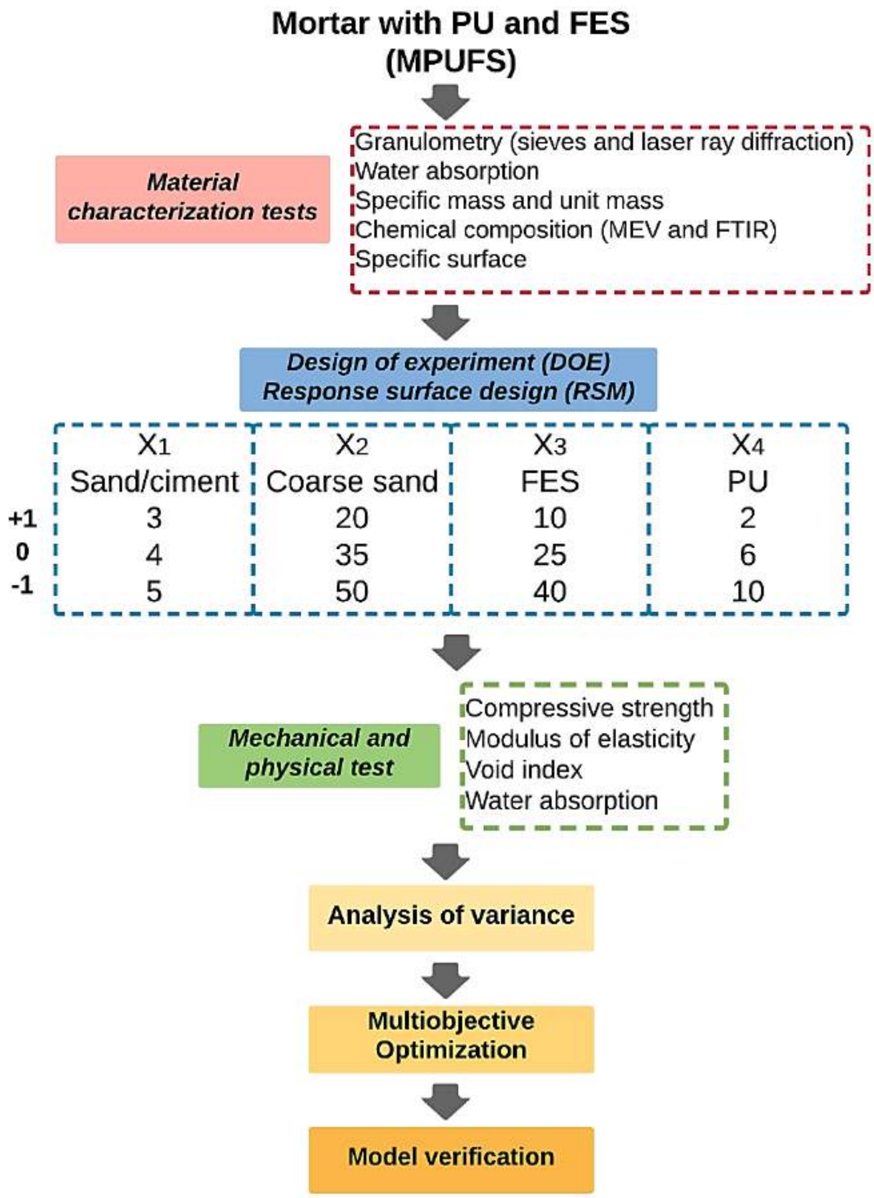

Figure 4-4: The schematic of the entire procedure.

Table 4-3: Parameters level in RSM BB model

Responses	Symbol	Parameters level		
		-1	0	+1
S/C	x_1	3.0	4.0	5.0
CS	x_2	20%	35%	50%
FES	x_3	10%	25%	40%
PU	x_4	2%	6%	10%

In Table 4-3, S/C represents two families of 1:3 to 1:5 (read, three parts binder to five parts sand) mixtures, with the binder consisting of cement plus hydrated lime in the ratio of 4:1, i.e., four parts cement and one part lime. The CS represents the percentage of coarse sand replacing fine sand from 20% to 50%, where coarse sand has a particle size ranging from 1.2mm to 4.8mm and fine sand from 0.75mm to 1.2mm; FES mass replaces NS from 10% to 40%; PU mass replaces NS from 2% to 4%.

The water to cement ratio (w/c) in the 1:3 mixes was equal to 0.45 and for the 1:5 mixtures it was 0.55. For the 1:4 center points the water to cement ratio was 0.50. The superplasticizer content was 1.5 of the cement weight for all mixtures. With these proportions were obtained spreads in the fresh state of 260 ± 20mm recommended by the standard [165] for coating mortar use that was taken as the limit for control of the study. The total number and experiment is obtained from Eq 4-1.

$$N = 2^k + 2k + c \qquad \text{Eq 4-1}$$

Where k is the number of variables studied, 2^k represents the factorial points of all combinations of the coded values ranging from ± 1 at the corner of the cubes; 2^k expresses the axial points at a distance of ± α from the origin; c is the number of trials at the central point in which all coded values are at level 0 [166]. According to Eq 4-1, 25 points of experiments were suggested for the 4 factors, at all points the experiments were replicated 3 times.

The Table 4-4 shows the experiments planned by the BB RSM in non-randomized to ascending order. The variable S/C represents the proportions of amount of sand (fine aggregate) per cement used in the mixture, for example in M_1 there are 3 portions of sand for 1 of cement. The variable SC represents the proportion of coarse sand (retained on the 1.2mm sieve) in relation to fine sand (passing the 1.2mm sieve), for example in the M_{17} mixture there is 35% coarse sand and 65% fine sand. The variables FES and PU represent the proportions of foundation sand from the exhaust and polyurethane that replaces the natural sand in mass, for example in the M5 mixture there is 10% FES and 2% PU and 88% natural sand. The Table 4-5 shows the consumption of the inputs used for each mixture per 1000 cm^3 of material. For better organization Table 4-4 was separated into three parts, the first of the mixtures with consumption of 329.67g and w/c ratio of 0.55, the second with consumption of 385.02g and w/c ratio of 0.50, the third with consumption of 462.71g and w/c ratio of 0.45.

Table 4-4: Experimental planning performed on the RSM BB

Mixtures code	S/C	SC	FES	PU
M_1	3	20%	25%	6%
M_2	5	20%	25%	6%
M_3	3	50%	25%	6%
M_4	5	50%	25%	6%
M_5	4	35%	10%	2%
M_6	4	35%	40%	2%
M_7	4	35%	10%	10%
M_8	4	35%	40%	10%
M_9	3	35%	25%	2%
M_{10}	5	35%	25%	2%
M_{11}	3	35%	25%	10%
M_{12}	5	35%	25%	10%
M_{13}	4	20%	10%	6%
M_{14}	4	50%	10%	6%
M_{15}	4	20%	40%	6%
M_{16}	4	50%	40%	6%
M_{17}	3	35%	10%	6%
M_{18}	5	35%	10%	6%
M_{19}	3	35%	40%	6%
M_{20}	5	35%	40%	6%
M_{21}	4	20%	25%	2%
M_{22}	4	50%	25%	2%
M_{23}	4	20%	25%	10%
M_{24}	4	50%	25%	10%
M_{25}	4	35%	25%	6%

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Table 4-5: Consumption of materials by mixing mortar for 1000cm³

	Cement(g)	Lime(g)	NS(g)	FES(g)	PU(g)	Sp(g)	w/c
M_2	329.67	82.42	1541.04	82.42	24.89	4.95	0.55
M_4	329.67	82.42	1541.04	82.42	24.89	4.95	0.55
M_{10}	329.67	82.42	1557.63	82.42	8.30	4.95	0.55
M_{12}	329.67	82.42	1524.44	82.42	41.49	4.95	0.55
M_{18}	329.67	82.42	1590.49	32.97	24.89	4.95	0.55
M_{20}	329.67	82.42	1491.59	131.87	24.89	4.95	0.55
M_5	385.02	96.26	1493.83	38.50	7.75	5.78	0.50
M_6	385.02	96.26	1378.32	154.01	7.75	5.78	0.50
M_7	385.02	96.26	1462.81	38.50	38.76	5.78	0.50
M_8	385.02	96.26	1347.31	154.01	38.76	5.78	0.50
M_{13}	385.02	96.26	1478.32	38.50	23.26	5.78	0.50
M_{14}	385.02	96.26	1478.32	38.50	23.26	5.78	0.50
M_{15}	385.02	96.26	1362.81	154.01	23.26	5.78	0.50
M_{16}	385.02	96.26	1362.81	154.01	23.26	5.78	0.50
M_{21}	385.02	96.26	1436.07	96.26	7.75	5.78	0.50
M_{22}	385.02	96.26	1436.07	96.26	7.75	5.78	0.50
M_{23}	385.02	96.26	1405.06	96.26	38.76	5.78	0.50
M_{24}	385.02	96.26	1405.06	96.26	38.76	5.78	0.50
M_{25}	385.02	96.26	1420.57	96.26	23.26	5.78	0.50
M_1	462.71	115.68	1251.49	115.68	20.96	6.94	0.45
M_3	462.71	115.68	1251.49	115.68	20.96	6.94	0.45
M_9	462.71	115.68	1265.46	115.68	6.99	6.94	0.45
M_{11}	462.71	115.68	1237.51	115.68	34.94	6.94	0.45
M_{17}	462.71	115.68	1320.90	46.27	20.96	6.94	0.45
M_{19}	462.71	115.68	1182.08	185.08	20.96	6.94	0.45

4.2.3 Sample Preparation and Test Procedures

First, the dry materials, cement, aggregates were mixed with two thirds of the total volume of water and then mixed for 2 min. Then the remaining water and the superplasticizer were slowly added and mixed for another 4 min. Next, consistency index test was performed on MPUFS in the fresh state according to the standard [165]. Finally, the fresh MPUFS was placed in the molds for the physical and mechanical tests, respectively. The samples remained in an environment with relative humidity higher than 90% for 24 h and then were submerged in drinking water in a curing tank with saturated lime water at 21 ± 3 °C.

The first two tests that were performed were the voids content and absorption by immersion, according to the prescriptions of [116], for mortar with 28 days of age, using specimens with 50 mm in diameter and 100 mm in height. Three samples were molded for each mortar mixture developed.

The following procedures were (a) Immersion of the samples in water at 20 ± 3 °C until there was mass constancy over a 24 h interval, i.e., that the samples did not show an increase greater than 0.5%. This mass is denominated (m_i) and was determined on a hydrostatic balance; (b) The samples were removed from the water and with a damp cloth their surfaces were dried to remove surface moisture. The saturated masses of the samples were then determined (m_{sat}); (c) The sample was then dried in an oven at 105°C until successive determination of the masses over a 24 hours interval showed no reduction in mass greater than 0.5%. After removal from the oven, the samples were cooled to room temperature for subsequent determination of the oven-dry mass (m_S). The values of void index and absorption by immersion were calculated by Eq 4-2 and Eq 4-3, respectively.

$$Absorption = \frac{m_{sat} - m_s}{m_s} \times 100 \qquad \text{Eq 4-2}$$

$$Void\ index = \frac{m_{sat} - m_s}{m_{sat}} \times 100 \qquad \text{Eq 4-3}$$

The next test performed was the modulus of elasticity. The measured modulus of elasticity was the dynamic modulus of elasticity by the natural frequencies of vibration method according to the standards [167, 168] at ages of 28 days of curing [121]. This test is known as the Impulse Excitation Technique (TEI), because it was performed by excitation of the MPUFS samples. The steps for performing the tests were: (a) determination of the geometry and dimensions of the samples; (b) measurement of the mass of the samples; (c) characterization of the modulus of elasticity through the TEI using the Sonelastic® equipment.

Finally, compressive strength test was performed on cylindrical samples of MPUFS at the age of 28 days according to standard [169]. The samples were removed from the cure and remained in desiccator for 24 h, then were subjected to axial loading using Electro-Hydraulic Universal Testing Machine.

Finally, an interactive summary of the sequence of execution of the experiments performed in this study is shown in Figure 4-5.

Figure 4-5: Sequence of experiments to perform the MPUFS.

4.3 Experimental observations and discussion

The average physical and mechanical test results for the 25 MPUFS mix designs are illustrated in Table 4-6. Details and discussion of the results are provided in the following sections.

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Table 4-6: Average response for each measured property of the MPUFS at 28 days of curing

Mixtures	f_{ck} (MPa)	E (GPa)	*e*(%)	Wa(%)	Sp (mm)
M_1	26.2	20.4	5.6	5.0	293.1
M_2	10.3	10.3	5.0	7.8	263.3
M_3	25.1	18.9	7.3	6.7	291.7
M_4	10.3	9.5	4.7	8.3	262.7
M_5	19.9	18.3	7.5	8.0	279.9
M_6	19.8	19.5	7.4	8.3	285.1
M_7	18.1	16.0	9.7	9.9	243.5
M_8	16.8	16.1	9.5	10.2	259.4
M_9	23.6	23.3	5.5	5.7	290.4
M_{10}	9.1	11.4	7.1	8.1	270.5
M_{11}	22.3	20.0	7.6	8.4	266.0
M_{12}	8.8	8.7	9.2	9.9	247.5
M_{13}	21.1	15.8	5.4	7.5	269.8
M_{14}	20.0	15.7	5.2	9.3	272.8
M_{15}	19.7	15.8	5.4	7.6	279.4
M_{16}	19.3	15.4	5.2	9.3	287.0
M_{17}	24.2	19.8	7.6	7.7	280.3
M_{18}	9.5	8.6	9.5	10.3	253.4
M_{19}	23.9	20.5	7.4	7.8	292.3
M_{20}	9.5	8.3	9.3	10.1	262.1
M_{21}	21.1	20.6	3.1	5.1	288.6
M_{22}	20.9	19.1	3.2	6.7	276.6
M_{23}	18.6	14.5	5.3	7.2	254.2
M_{24}	17.9	14.2	5.2	8.9	258.2
M_{25}	22.3	21.7	1.0	1.3	321.0

4.3.1 Pareto chart

The Pareto chart shows the absolute values of the standardized effects from largest effect to smallest effect, so a vertical reference line is positioned to indicate which effects are statistically significant in the analyzed process. The reference line for statistical significance depends on the level of significance called alpha (α) [170]. Since the backward elimination option was used in this study, the significance level used by Minitab is the significance level of the term removed from the model, known as alpha to remove.

The use of the Pareto Chart determines the importance and magnitude of a variable or effect on the final response in a hierarchical manner. Thus, to state whether a variable is important and should be used in the model, it is enough to analyze which variables crossed the reference line. Because the Pareto Chart displays the absolute values of the effects, you cannot determine which effects increase or decrease the response, only that they affect the response, so to perform this analysis you can use the regression coefficient generated in the [171]. This will be analyzed in the next section.

From Figure 4-6a it is possible to state that the Standardized Effect is 1.67, the linear effects (D, A, B) of the MPUFS affect compressive strength (f_{ck}). All tested quadratic effects (CC, DD, AA, BB) of the MPUFS significantly affect compressive strength and the order of magnitude is the same as shown in Figure 4-6a, i.e. linear PU is more important, followed by quadratic FES terms and so on. The modulus of elasticity (E), Figure 4-6b, is affected by the linear variables (D, A, B) and the quadratic variables (CC, DD, AA, BB) and following a very similar behavior to that of the compressive strength.

The voids content (*e*), Figure 4-6c, is most affected by quadratic variables (CC, AA, DD), followed by the linear variables (D, A). What is new is that it is also affected by the interaction of two AB variables, i.e., the interaction of S/C and SC causes important effects on the MPUFS. The water absorption (W_a), Figure 4-6d, behaves similarly to the voids content. However, there are more effects that affect the absorption and do not affect the voids index which are quadratic effect (BB), linear effect (B) and a new interaction (AD).

The fresh state of the MPUFS was assessed by the scattering test which is shown in Figure 4-6e. In this way it is possible to understand that the most significant effects DD and D, quadratic and linear, respectively, originate in the PU. In general, all the variables involved significantly affect the behavior in the fresh state, with PU being the most significant factor, followed by S/C, FES and SC.

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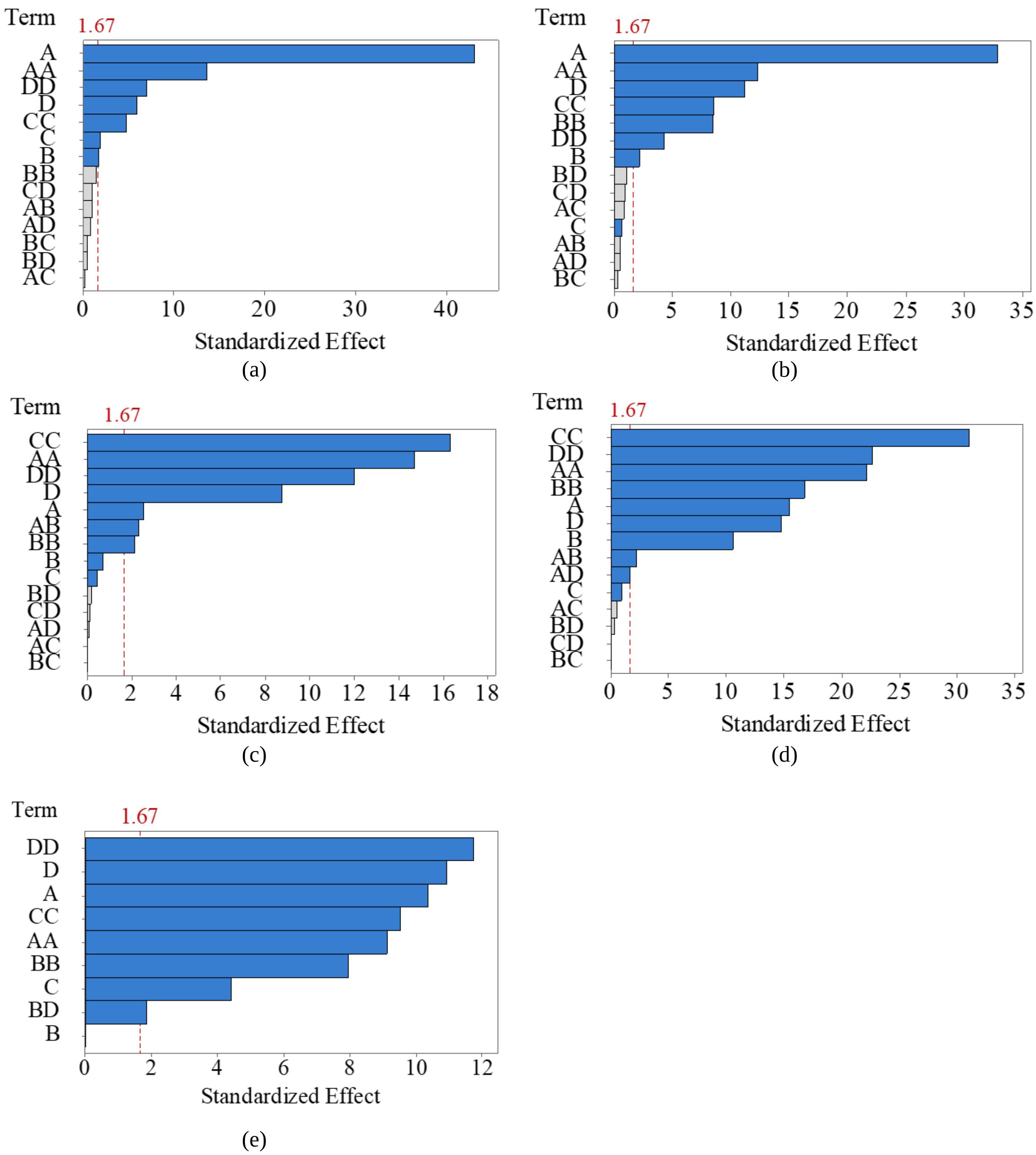

Figure 4-6: Standardized effect for MPUFS. (a) compressive strength; (b) modulus of elasticity; (c) void index; (d) water absorption; (e) Spreading.
Legend: A: S/C; B: SC; C: FES; D: PU

4.3.2 Analysis of variance (ANOVA)

The ANOVA test was used to statistically determine the significance of the variables studied in the MPUFS. In ANOVA the total sum of squares expresses the total variation that can be attributed to multiple factors and is defined as the combination of treatment sum of squares (SST) and Sum of Squares of the residual Error (SSE) [166].

Thus, ANOVA is a method often used to evaluate the influence of different variables on the results of experiments.

The results of the ANOVA are given in Table 4-7. According to the results, the lack of fit p-value for the factors (S/C, SC, FES, PU) that were below 0.005 confirms that these factors have significant influence on the responses. The adopted model was the full quadratic model, its levels can be seen in Table 4-3. From Table 4-7 it can be seen that only for linear X_3 (FES) there was no significance. In order to optimize the model, the back elimination option was selected, which allows only significant factors in the model. The regression coefficient describes the direction and size of the relationship between the variable factor and a predicted response. Coefficients are the numbers by which the values of the terms in the regression equations are multiplied [172].

Considering the interactions of the first and second order terms, the following second order polynomial response surface Eq 4-4 was implemented [173]:

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_i x_i^2 + \sum_{i<1}^{k} \beta_{ij} x_i x_j + e(x_i, x_j, ..., x_k) \qquad \text{Eq 4-4}$$

Where y is the answer (f_{ck}, E, *e*, W_a); X_i and X_j is an independent variable (C/S, SC, FES, PU); b_0 is a constant; b_i, b_{ii} e b_{ij} are the first and second order regression and interaction coefficients respectively; k is the number of significant factors; *e* is the error (including test and regression errors).

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Table 4-7: The results of ANOVA for MPUFS

Term		Fck (MPa)	E (Gpa)	*e* (%)	Wa (%)	Sp (mm)
Constant	Coef	21.576	21.678	0.970	1.307	321.020
	p-value	< 0.000	< 0.000	0.024	< 0.000	< 0.000
X_1	Coef	-7.315	-5.497	0.305	1.099	-12.850
	p-value	< 0.000	< 0.000	0.014	< 0.000	< 0.000
X_2	Coef	-0.293	-0.373	0.084	0.753	0.030
	p-value	0.089	0.029	0.492	< 0.000	0.981
X_3	Coef	-0.314	0.109	-0.056	0.068	5.470
	p-value	0.069	0.517	0.647	0.340	< 0.000
X_4	Coef	-0.995	-1.878	1.057	1.045	-13.530
	p-value	< 0.000	< 0.000	< 0.000	< 0.000	< 0.000
X_1*X_1	Coef	-3.633	-4.245	3.666	3.242	-23.350
	p-value	< 0.000	< 0.000	< 0.000	< 0.000	< 0.000
X_2*X_2	Coef	-	-2.940	0.527	2.455	-20.340
	p-value	-	< 0.000	0.038	< 0.000	< 0.000
X_3*X_3	Coef	-1.258	-2.964	4.070	4.522	-24.360
	p-value	< 0.000	< 0.000	< 0.000	< 0.000	< 0.000
X_4*X_4	Coef	-1.853	-1.484	2.981	3.315	-30.020
	p-value	< 0.000	< 0.000	< 0.000	< 0.000	< 0.000
X_1*X_2	Coef	-	-	-0.484	-0.283	-
	p-value	-	-	0.024	0.025	-
X_1*X_4	Coef	-	-		-0.207	4.010
	p-value	-	-		0.096	0.066

4.3.3 Main effect of plots

The main effect of plots shows the individual action of each study variable on the analyzed properties, the curves were generated only for the properties that were considered in the model. According to Figure 4-7 the region of each variable is close to a curvature region, i.e., maximum values of compressive strength and modulus of elasticity; as well as minimum values of absorption and void index were reached for approximately S/C equal to 3.50, SC equal to 35, FES equal to 25% and PU of 3%. This was only possible thanks to the methodology adopted in this study that first runs the full FFD to determine the curvature ranges for each variable and then applies the RSM to study the behavior exactly in these curvature ranges.

The compressive strength values range from 8.8 MPa to 26.2 MPa, Figure 4-7a and modulus of elasticity range from 8.3 GPa to 23.3 GPa, Figure 4-7b. It is expected that the higher S/C the lower strength the MPUFS will have, because, it will have less binder (cement) [174]. However, this does not happen in a linear manner, with mixtures with S/C equal to 3.50 having slightly higher strengths than S/C equal to 3.0. This probably occurs because mixtures with higher cement contents have higher heat of hydration and greater drying shrinkage that can cause micro cracks and decrease the final strength [175]. Another factor affecting strength and modulus of elasticity is particle packing which is most likely influenced by SC. The particles obtained better dimensional arrangement with SC contents equal to 35 of coarse sand which is very close to that used in many conventional concrete mixtures [176].

The FES acts as a very fine aggregate, because it has an intermediate granulometry between cement and NS, Figure 4-1. Thus, FES acts as a nucleation and packing agent for the larger particles, the author [161] found similar result of FES in concrete. This can be explained by the fact that 50% of the FES particles are smaller than 150 µm and have round shape as can be seen in SEM [177].

It is expected that the incorporation of PU in mortars and portland cement-based concretes presents a loss of mechanical strength, several studies have shown this behavior [132]. However, it can be seen in Figure 4-7a and Figure 4-7b that for small contents of 3% to 4% no significant loss of mechanical properties occurs. The mechanical strength and impermeability of cementitious composites is greatly affected by the presence and distribution of capillary voids larger than 50 nm called macro-pores. While the voids smaller than 50 nm, called micropores that play an important role in drying shrinkage and creep [174]. In this way it is likely that the PU in percentages of 3 to 4% was able to penetrate these macro-pores. Although PU has low resistance, its presence is preferable to that of voids that offer no resistance, slightly reducing the loss of resistance.

The results for absorption, Figure 4-7c, and void index, Figure 4-7d, show a tendency to minimize in the suggested range. The only factor that did not influence significantly was the SC in the voids index. Results found by other authors such as [132] show a tendency to increase absorption and void index in the presence of aggregates of polymeric origin. In this sense Figure 4-7c and Figure 4-7d show this tendency of increase in the voids index and absorption for contents higher than 4% PU and 25 of FES.

However, this trend is not linear and as shown in Figure 4-7c and Figure 4-7d there is an optimal inflection point located at the center of curvature.

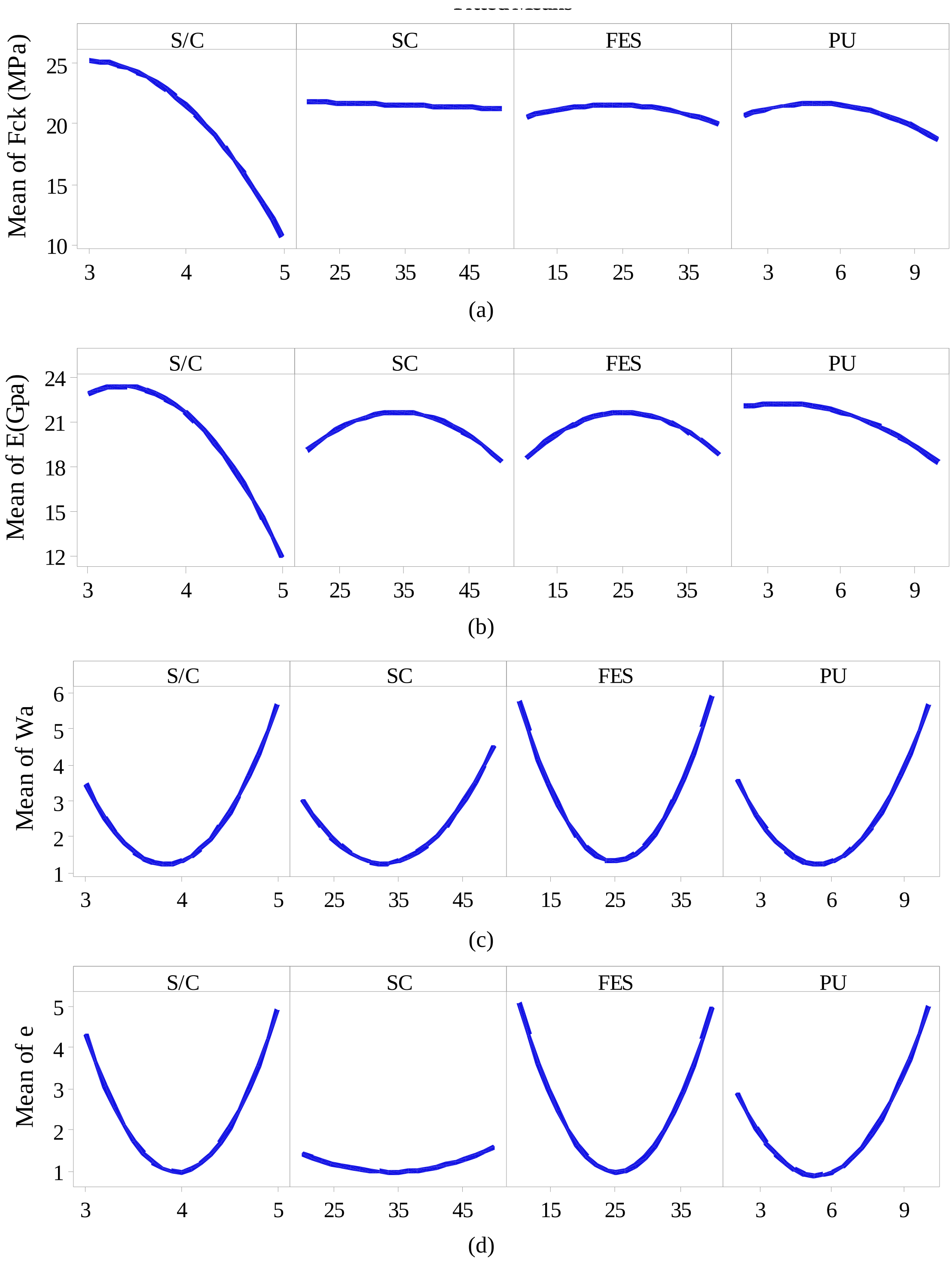

Figure 4-7: Main Effects Plots for MPUFS samples. a) compressive strength; (b) modulus of elasticity; (c) void index; (d) water absorption.

From Eq 4-5 to Eq 4-8 were obtained using the quadratic RSM-BB model used in this study. The R^2 is expressed as a percentage between 0 to 100, with 100 representing perfect correlation and zero representing no correlation. The R^2(adj) is a modified version of R-Squared that adds precision and reliability by considering the additional impact of independent variables that tend to bias the results of R^2 measurements.

The degree of regression model fit can be assessed by means of the variance ratio (R^2) and the difference between R^2(adjustable) and R^2(predicted) should not be greater than 20% [178]. In

Table 4-8, the difference between R^2(adjustable) and R^2(predicted) is presented, and it can be seen that the difference between R^2(adjustable) and R^2(predicted) for all responses (f_{ck}, E, *e*, W_a) were less than 20%, revealing that the model for all responses is adequate with reality and the generated equations can be used to predict responses with adequate accuracy.

$$f_{ck} = -0.201 + 1.947x_1 + 0.01383x_3 + 0.0641x_4 - 0.3013x_1x_1 - 0.000299x_3x_3 - 0.006446x_4x_4 \quad \text{Eq 4-5}$$

$$E = -1.668 + 2.131x_1 + 0.04642x_2 + 0.03784x_3 + 0.0269x_4 - 0.3148x_1x_1 - 0.000685x_2x_2 - 0.000752x_3x_3 - 0.00465x_4x_4 \quad \text{Eq 4-6}$$

$$e = 24.68 - 8.505x_1 - 0.1568x_2 - 0.25420x_3 - 0.6583x_4 + 1.0896x_1x_1 + 0.002551x_2x_2 + 0.005075x_3x_3 + 0.05852x_4x_4 - 0.00525x_1x_2 \quad \text{Eq 4-7}$$

$$Wa = 21.724 - 6.705x_1 - 0.22164x_2 - 0.23352x_3 - 0.5883x_4 + 0.8804x_1x_1 + 0.003474x_2x_2 + 0.004681x_3x_3 + 0.05560x_4x_4 - 0.00364x_1x_2 - 0.01126x_1x_4 \quad \text{Eq 4-8}$$

Table 4-8: Regression model variation (R^2)

Responses	R^2	R^2adj	R^2pred	R^2 adj - R^2 pred
Compressive strength - Fck	98.16%	98.00%	97.77%	0.23%
Modulus of elasticity - E	96.19%	95.73%	95.16%	0.57%
Void index - e	94.13%	93.31%	91.85%	1.46%
Water absorption - Wa	98.40%	98.15%	97.79%	0.36%

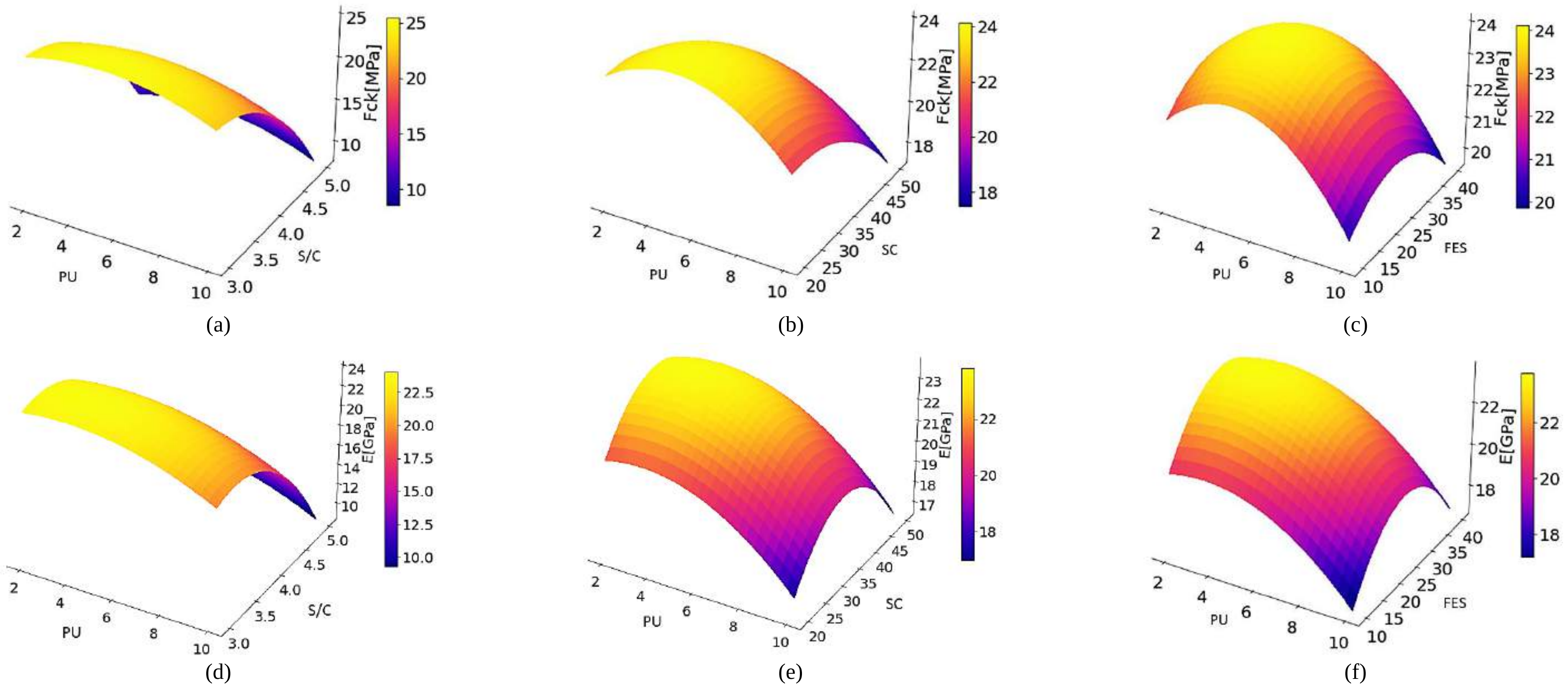

Figure 4-8: Influence of the variables studied on Portland cement mortars. (a) - (c) Compressive strength; (d) - (f) Modulus of elasticity.

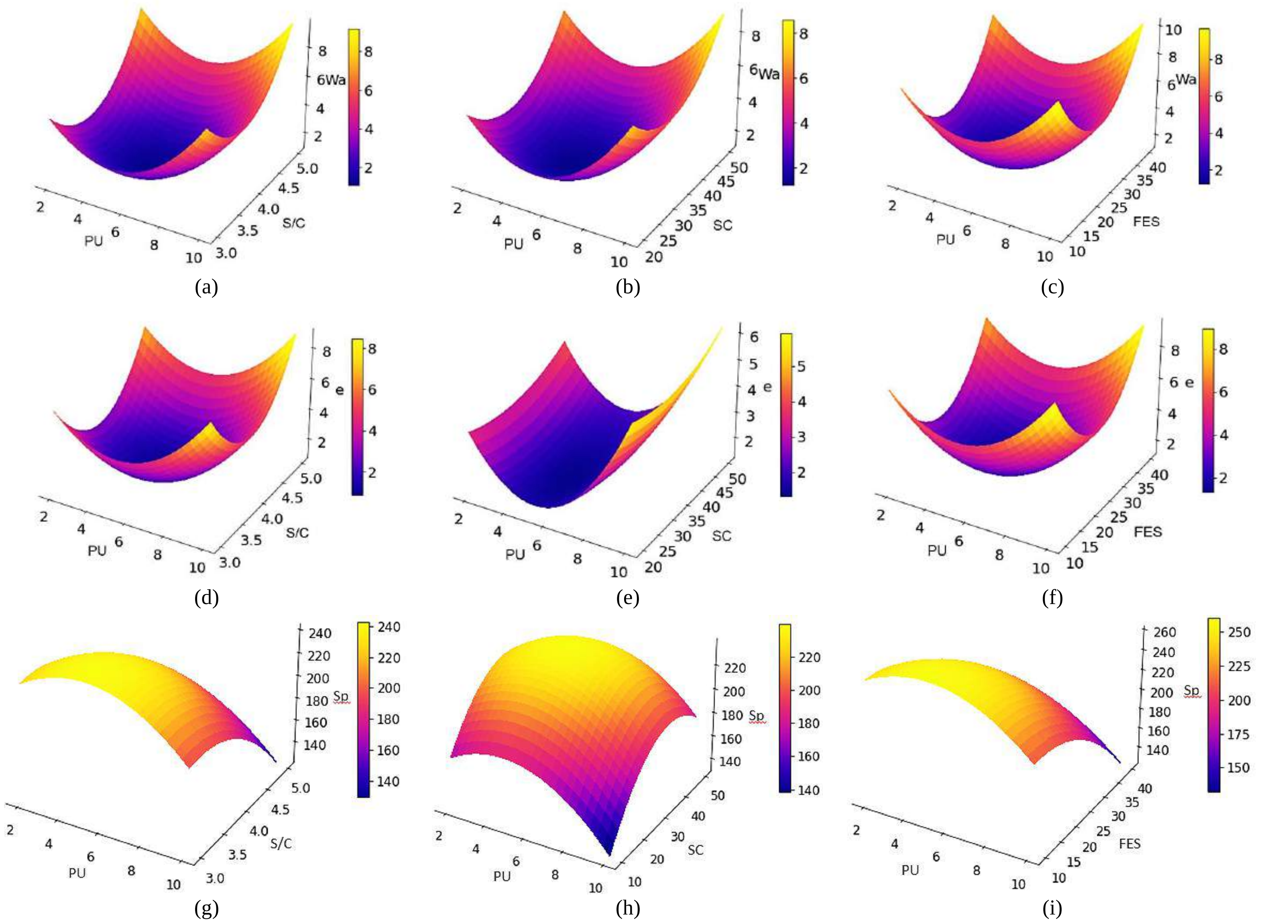

Figure 4-9: Influence of the variables studied on Portland cement mortars. (a) to (c) Absorption by immersion; (d) to (f) Voids index; (g) to (i) spreading.

To better understand the influence of the trend variables on the processing responses, 3D surface plots were generated and are presented in Figure 4-8 and Figure 4-9. In the surface plot, the responses were plotted on the "z" axis and the process variables on the "x" and "y" axis. From of elasticity for all the tested cases shows varying degrees of bending. That is around 3% to 5% PU, 3.50 S/C, 25% to 30% SC and 25% FES. Thus, the mechanical resistance at these points is the maximum possible, and with increases in the incorporation of PU or FES the mechanical resistance decreases considerably.

An analysis in Figure 4-9(a)-(f) shows that both the absorption and the void ratio have curvatures in the tested point ranges. The Absorption, Figure 4-9(a)-(c) is lower for PU equal to 4% to 7%, S/C equal to 3.50, SC equal to 25% and 40% and FES equal to 20% to 30%. A similar result is found for the voids index, Figure 4-9(d)-(f), which only when comparing PU with SC is a little different, as the percentage of PU found for the minimum voids index has a limited range of approximately 5 to 6% PU. These results are proportional to those found in Figure 4-7 where a curvature occurs at the highlighted points. The Figure 4-9 collaborates with the results of Figure 4-7, in this sense the presence of PU modifies the fresh state. Thus, according to Figure 4-7, for a percentage greater than 4% of PU, a decrease in workability occurs in all observed combinations. A percentage varying between 3% and 4% is the ideal amount of PU to promote greater spreading.

It is important to highlight the non-linear nature of the responses obtained, which can be seen by the sharp curvature of most of the variables investigated. This indicates that the quadratic model adopted (RSM-BB) is probably better suited to predict their behavior. To be sure of this, in the next sections this hypothesis will be tested.

4.3.4 Multiobjective Optimization

At this point of the study, it is possible to obtain the optimization of the mix designs for MPUFS, after the validation of the proposed model for compressive strength, modulus of elasticity, voids index, water absorption and spreading. Thus, the optimal proportions of S/C, CS, PU and FES used in the making of MPUFS can be determined based on the analyzed properties.

Five scenarios were chosen to optimize the properties of the MPUFES. To optimize the properties, a multi-objective optimization model was chosen in which multiple variables are modified simultaneously to achieve an optimal mixture design, targeting each pre-established objective.

The Table 4-9 shows the configuration for the optimization. The first scenario chosen was to maximize $F_{c,k}$ and E, and minimize *e*, W_a. The second scenario was chosen to only maximize the $F_{c,k}$ and the third scenario was chosen to minimize the *e*, W_a. The fourth scenario was the maximization of spreading. The fifth scenario sought to optimize the proportion of mortars with the minimum compressive strength required by the Brazilian standard [121]. Also, in the fifth scenario the FES and PU variables were set at a maximum of 40% for FES and 10% for PU to ensure maximum waste usage and minimum resistance. Note that all five optimized scenarios have some justified practical relevance. For example, maximum strength and minimum absorption are desired qualities in mortars.

Table 4-9: Multiobjective optimization of MPUFS.

Settings to optimize	Optimum mixture design			
	S/C	SC	FES	PU
max($f_{c,k}$; E) min(e; W_a)	3.81	30.30	25.20	4.75
max($f_{c,k}$)	3.61	28.48	25.15	4.34
min(*e*, W_a)	3.81	32.42	24.85	5.31
max(Sp)	3.72	34.55	26.67	5.10
Target($f_{c,k}$: 2MPa)	4.89	50.00	40.00	10.00

Through an evaluation of the values obtained in the optimization in the Table 4-9, it is possible to verify that the values follow a pattern. For example, for S/C the proportion of sand to cement was always between 3.61 and 3.81, that is, a variation of 5.3%. For SC, which represents the amount of coarse sand, there is a variation from 28.48 to 34.55, that is, a variation of 17.6%. For FES there is a variation from 24.85 to 26.67 which corresponds to a percentage variation of 6.8% and finally for PU there is a variation from 5.31 to 4.34 which corresponds to a percentage variation of 18.3%. In general, the variation of proportions to reach the optimal content that is desired in most applications varies at most 18.3%, value found for PU. The last row of Table 4-9 shows the optimization of the fifth scenario. In this case there is a difference between the first four scenarios, because there is a greater use of FES and PU waste in detriment of resistance.

4.3.5 Model verification

The equations were developed to predict the responses of a set of mixtures developed within the range of variables investigated. Thus, this section is designed to verify the equations developed in RSM BB. For this purpose, the mixtures predicted in Table 4-9 that refer to the multi-objective omissions were selected. These mixtures were subjected to the same tests as the initial samples, and the tests were prepared according to the same procedures cited in the methodology section. Table 4-10 shows the proportions of the test mixtures and the comparison with the responses from the model developed by RSM BB. The validation of the developed regression equations will be performed by calculating the absolute relative derivation Δ(%) given by Eq 4-9. For the purpose of validation of the equations the value of Δ(%) should be less than 10% [179].

$$\Delta(\%) = \left(\frac{Experimental - Model}{Experimental} \right) * 100 \qquad \text{Eq 4-9}$$

Table 4-10: Comparison between RSM predicted in the model and actual values

Scenarios	RSM	Exp	Δ(%)	RSM	Exp	Δ(%)	RSM	Exp	Δ(%)	RSM	Exp	Δ(%)	RSM	Exp	Δ(%)
	$F_{c,k}$ (MPa)			E (GPa)			e (%)			Wa (%)			Sp (mm)		
1st	47.05	43.85	-7.30	31.16	30.68	-1.56	1.00	1.05	4.76	1.19	1.10	-8.18	-	-	-
2nd	47.43	49.05	3.30	-	-	-	-	-	-	-	-	-	-	-	-
3rd	-	-	-	-	-	-	0.94	1.05	10.48	1.06	0.98	-8.16	-	-	-
4th	-	-	-	-	-	-	-	-	-	-	-	-	324.63	305.05	-6.42
5th	2.00	1.79	-10.50	-	-	-	-	-	-	-	-	-	-	-	-

RSM: value predicted by the model; Exp.: experimental value; Δ(%): absolute relative deviation.

As shown in Table 4-10, the results for Δ(%) are all below to 10%, which indicates that the model accuracy is acceptable within the established parameters.

4.4 Conclusions

In this study, RSM was developed to obtain statistical equations to predict the properties of compressive strength, elasticity model, void index, and water absorption of MPUFS. For this purpose, a BB model of RSM was developed that allowed the use of a quadratic model for each response at 3 levels and 4 control factors that included S/C, SC, FES, and PU. Based on the results, the following conclusions can be drawn:

Comparing the predictions of the RSM and the test results, it appears that the RSM can predict the mechanical and physical strength properties of mortar as MPUFS. The positive effect of PU can be observed in percentages varying from 2 to 3% of substitution by mass of fine aggregate. Under these conditions, stability in the compressive strength and modulus of elasticity is obtained, as well as a decrease in the void index and water absorption. The positive effect of FES can be observed in percentages close to 25% of replacement in the mass of fine aggregate. Under these conditions, an improvement in mechanical strength and modulus of elasticity is obtained, as well as a decrease in the void index and water absorption. It was found that the studied S/C and SC factors can be used to optimize the properties of MPUFS. The positive effects of S/C and SC are observed for values of 1:3.0 to 1:3.5 and approximately 0.35, respectively, where the analyzed properties find their best results. The properties in the fresh scattering state are influenced by the variables S/C, SC, PU, and FES analyzed in this study. In order to achieve the greatest spread and, consequently, better workability, the proportions should be approximately the following: S/C 3.72, SC 34.55, PU 26.67, and FES 5.10.

According to the optimization of the results, FES and PU could be used in mortars, finding satisfactory results in compressive strength and modulus of elasticity as well as absorption index and void content that indicate better durability. In this way, it could be possible to obtain a reduction of some environmental impacts related to the inadequate disposal of this waste in the environment. According to the optimization results, we could use 40% PU and 40% FES to produce a mortar with a minimum compressive strength of 2.00 MPa. This field of research still needs to be further explored. aiming to balance the demand of the civil construction industry with environmental issues such as waste disposal. As a result, it is critical that future research investigates other methods of response prediction. well as implementation of properties of practical interest in the application of cementitious composites in construction, such as dynamic responses in structures, vibrations and not only static responses as in this study.

5 AN EXPERIMENTAL DYNAMIC STUDY OF CEMENT MORTAR WITH POLYURETHANE RESIDUES AND FOUNDRY SAND

Although many studies have reported on the application of experimental, statistical, numerical, and computational tools to composite structures, few have focused on the use of analysis of variance (ANOVA) to analyze experimental data and Artificial Neural Network (ANN) as a technique to predict the modal responses of Portland cement mortars. In this study, the modal responses of Portland cement mortars with polyurethane waste and exhausted foundation sand were examined using Design of experiments (DOE) and Artificial Neural Network (ANN). The studies used free vibration, and the responses that were studied were natural frequency and damping factor. According to the experimental findings, the combined PU and FES waste can drastically alter the mortars' inherent frequency and damping. Depending on the amount of waste used, this change can range from a rise of 11.6% to a decrease of 21.7% when compared to the reference samples. The ANN demonstrated a strong connection in predicting the experimental findings after being trained with the experimental data.

5.1 Introduction

One of the biggest challenges for civil engineering today is the production of high-performance materials without producing significant waste or that incorporate recycling materials in their composition. However, in the last decades the target of the production of materials was only the performance, thus the environmental issue related to the waste of its production was neglected. This has resulted in the rampant production of waste of various kinds such as plastics, wood, cement, for example, in the last decade were produced approximately 1.00 and 1.13 billion tons of construction waste in the U.S. and China, respectively [180]

Therefore, research that aims to performance characteristics of materials and at the same time use recycled materials is important. This study was based on this premise, the wastes explored in this study are polyurethane (PU) and exhaust casting sand (FES) which are of different physical and chemical nature but which impact the environment in an equivalently negative way [181]. The foundry sand, on the other hand, is a by-product of metallurgical production, the so-called function sand or green sand is incorporated into molds that can be reused several times during the production of metal parts [8, 9]. During part manufacturing, factory exhaust fans capture high surface area sand residue that comes loose in the molds. FES is the airborne residue that comes loose from molds and can be captured by exhaust fans. Thus, FES is generally finer than conventional exhaust sand [11]. And because it is thinner, its disposal in the environment is difficult due to the numerous possibilities of contamination to the local fauna and flora [10]. The PU originates from the polymer family, so it is very versatile and can come in many shapes and sizes and be used on a large industrial scale [182]. This product has a low production cost and high durability, and is widely used in the construction sector and in the household appliance trade in general [4, 183].

One of the characteristics that need to be studied in cementitious mortars and concrete are the dynamic properties, or how this type of material behaves under dynamic loading. In this way, modal analysis is a technique for examining the dynamic properties of structures. Modal analysis is often performed by computer analysis and experimental modal analysis (EMA) in this way it is able to distinguish vibration modes, natural frequencies and damping rate [184].

Some recycled materials have natural advantages regarding mechanical energy dissipation and high damping rates. One of these characteristics is the presence of a weak ITZ that decreases the friction and sliding behavior between each internal of the material [185]. Another characteristic is uniform particle size distribution and compact form factor that can provide better vibration energy distribution. Finally another characteristic is the low specific mass and large voids that provide better vibration energy dissipation [186].

The very good damping characteristic of cementitious materials using waste shows the potential for application of this environmentally friendly material in structural vibration control [187], for instance, on the basis of subway or high-speed trains [188, 189]. Nevertheless, the implementation of these technologies to achieve better vibration control still requires a progress on two key problems: a better energy-efficient concrete and a process to build the gap between the damping properties of concrete materials and their elements and structures [190].

Reinforced concrete structures that are made of cementitious materials are inevitably subjected to dynamic loads. These loads may be part of their use and occupation such as vehicular traffic on a bridge, pedestrians walking along a walkway or stairway. Other loads that are significant, such as wind, earthquakes, and explosions that vary in intensity during the life of a building and cause dynamic responses in the structure [191]. Damping is an essential parameter for determining the dynamic response of a structure under various possible loads and determining the degree of damage before low to medium intensity events occur [192]. It is widely accepted that damping represents the energy dissipation capability of a material or structure. However, due to the complex nature and mechanisms of multiple responses, modeling damping behavior is still an active area of study [190].

The reason that damping behaviors cannot properly be explained by a unique universal damping model is that the damping of concrete materials is composed mainly of viscoelastic damping and hysteresis damping [193]. For concrete materials, both the viscoelastic damping model and the complex damping model are widely used in assessing their viscous damping [194]. For concrete materials, the viscous damping model that considers the energy dissipated by various sources is commonly applied in structural design and analysis, and the equal viscous damping ratio is used to depict the damping capacity of concrete structures [195]. For the majority of cases, it is reasonable and acceptable to apply viscous damping and the associated equivalent viscous damping ratio in describing the internal damping characteristics of the concrete material and structure [196].

The incorporation of alternative materials with high damping properties, such as polymers, has been considered to promote the energy dissipation capacity of cementitious materials. This is done by considering their high flexibility and viscous damping mechanism [197]. Since the 1990s research has been done related to the topic. The first ones to discover the behavior of rubberized concrete and they have found that it exhibited lower compressive and tensile strength than natural coarse aggregate due to the low modulus and high Poisson's ratio of rubber [198]. In addition, the incorporation of rubber crumb varied the failure characteristics from brittle to ductile and plastic failure. A study seeking the optimization and improvement of the damping properties of concrete with polymeric materials was performed, in which analyzed the final microstructure of the material [199]. Compared with the natural aggregate, the dynamic performance of concrete gets significant improvement with the incorporation of polymeric nature products, contrary to what happens with the mechanical performance. Xue and Shinozuka (2013)[200] found results of 60% improvement in 20% of substitution, Moustafa and ElGawady (2015)[201] 35% in 30% of substitution, Zheng et al., (2008) [202] that this improvement can reach up to 140% in 45% of substitution. The equivalent viscous damping ratio of concrete columns (columns) with rubber obtained 13% and 150% improvement in dissipation energy [203].

The research related to concrete damping with cementitious materials is still in its early stages. But with the results already achieved it is safe to say that the incorporation of polymeric materials can improve the damping performance, flexibility and energy dissipation of cement elements [204]. According to Liang Fu (2022) [184], the improvement of damping can happen through a correct combination of coarse aggregate, fine aggregate, and polymeric material.

Regarding the dynamic properties, not only concrete with polymeric waste but also waste of various natures have been researched as a partial substitute for natural aggregates. Conducted reverse cyclic stress tests on beam-column connections and found that as the axial compression ratio increases, both the energy consumption and ductility decrease substantially [205]. Using recycled concrete blocks demonstrated that the building structure fully complies with seismic fortification standards using simulated vibration of the structure [206]. Clough R. W. and Penzien J. (2003)[207] performed creep tests, loading and unloading tests, and free vibration tests on reinforced RAC beams of varying ages and found that the damping characteristic of the RAC is related to the instantaneous recoverable deformation [207]. Concrete with recycled aggregate structure's basic mode shape has a lower natural frequency and a higher damping ratio than the concrete with natural aggregate structure [190, 208]. Studying the damping ratio of reinforced concrete structures Japan, identifies substantial damping ranges that can be achieved by structures [209]. An evaluation of the seismic resistance capacity of concrete with recycled material was performed by and proved that it has resistance [210]. In this study the author calculated an average equivalent viscous damping value of 0.217 at the ultimate load cycle using a low cycle load test on a recycled concrete column with a size ratio of 1:2.5. The investigated the seismic performance of concrete with recycled concrete columns after freeze-thaw cycles [211]. The experimental results indicate that at the same displacement level, the equivalent viscous damping coefficient of concrete with recycled column aggregate is slightly increased. Li et al., (2018)[211] found that the frequency of vibration of concrete is log-normal and that the damping ratio distribution is a superposition of two log-normal distributions. They measured the damping of recycled concrete using the free vibration dampening method. The dynamic modulus and nonlinear damping characteristics of concrete with recycled aggregate, as well as the static compressive strength and modulus of elasticity of concrete with recycled aggregate, using cyclic uniaxial compression experiments their results indicated that concrete with recycled aggregate had a higher loss factor than concrete with natural aggregate by [19].

The use of artificial neural network in cementitious materials has been explored to predict behavior in terms of mechanical strength, chemical reactivity and physical properties. Author Asteris et al., (2019)[212] makes the prediction of mortar strength based on its mixing components through artificial neural networks. The highly nonlinear relationship between the compressive strength of the mortar and the mixed components makes it difficult to predict strength. The study presents maps of compression resistance in order to facilitate the sizing of the mortar mixture.

Use of artificial neural network (ANN) to predict the compressive and flexural strength of a mortar made with modified zeolite additive (MZA)[213]. The input data used were six parameters: amount of cement, amount of silica sand, amount of modified zeolite additive (MZA), amount of water, curing period and load weights. The output data consisted of the compressive or flexural strength. The author concludes that ANN can be used to predict experimental results related to compressive and flexural strength.

Study the natural hydrated lime mortar and through a database uses three degrees of hydration, different aggregates and water content is performed by [212]. Artificial neural networks are used to describe the influence of mortar design on compressive strength. The study reveals that neural networks are effective in predicting the influence of input parameters used at different hydration ages.

Use of artificial neural network in corrosion modeling in cementitious mortars by the author [214]. It is known that many of the factors that affect corrosion are difficult to control. Thus, the artificial neural network can be a technique to be considered due to its ability to tolerate relatively imprecise, noisy or incomplete data, lower vulnerability to outliers, filtering capacity and adaptability. The study generates a corrosion current density prediction model using the artificial neural network approach. Several variables were used as input variables, namely: age, water/cement ratio, cement content, compressive strength, and type of kneading water, corrosion potential, solution resistance and polarization resistance. Finally, the resulting neural network model satisfactorily predicted the corrosion current density.

Based on the studies described, it is evident that the use of ANN to predict the behavior of cementitious materials is promising. Thus, the use of ANN to predict dynamic properties of mortars is justifiable. The authors highlight this as an innovation or novelty promoted by the present study.

In this study, the natural fine aggregate of Portland cement mortar was replaced by the combination of PU and FES to achieve the best configuration of energy dissipation capacity and damping properties. Thus, PU and FES were considered as partial substitutes for natural sand and the modifications of these variables were investigated. Other variables investigated were sand/cement S/C ratio and coarse sand SC content in the variation of properties. In this way, it is sought to produce an ecological mortar with relatively low cement consumption and reuse of natural resources. The modal data (natural frequency and loss factor) obtained from modal testing data were submitted to analysis of variance in order to evaluate its statistical significance. A design of experiments was performed considering a Response surface methodology (RSM) to obtain the best cement mortar manufacturing parameters. Finally, an artificial neural network is constructed to predict the modal properties based on manufacturing factors. Finally, the best designs from both methods were compared.

5.2 Theoretical background

5.2.1 Structural Vibration

Modal analysis can be defined as the study of the dynamic characteristics of a system independently of the applied loads and the respective responses of the structure or system. Thus the modal analysis identifies different modes of frequency, damping and deformation specific to each structure or system [215].

The modal analysis consists of evaluating the dynamic characteristics of a structure or system. These dynamic characteristics are known as modal shapes, damping, and natural frequency. According to the conditions of use/operation, structures are subjected to dynamic and/or static loads at the same time. That will respond to the excitations and the response must be quantified and analyzed within the defined design conditions. The observations of dynamic responses as a whole do not provide enough information to understand the structure or the system behavior, the modal analysis is configured as the best way to interpret the dynamic behavior of a structure [216].

According to the conditions of use/operation, structures are subjected to dynamic and/or static loads at the same time. That will respond to the excitations and the response must be quantified and analyzed within the defined design conditions. The observations of dynamic responses as a whole do not provide enough information to understand the structure or the system behavior, the modal analysis is configured as the best way to interpret the dynamic behavior of a structure [217, 218].

Natural frequency is the frequency at which a system will vibrate by itself, without external forces, after an initial perturbation. The number of natural frequencies of a system or structure can be obtained by the number of degrees of freedom of that system or structure. The vibration modes, on the other hand, are the deformations that each component should present when vibrating at its natural frequency. The decrease in oscillation of a system occurs due to the phenomenon of damping, which can be measured by the coefficient ζ, called damping ratio, which describes how oscillations decay after a disturbance [219].

The occurrence of damping results in energy loss from a system and the most common use for quantifying energy loss is the loss factor coefficient, denoted by η. The loss factor represents the lost energy (ΔE) by the potential energy at maximum displacement (U_{max}) Bottega W. J. (2006)[220] which is given in Eq 5-1. It is worth noting that when resonance occurs the loss factor is equal to twice the damping ratio.

$$\eta = \frac{\Delta E}{2\pi U_{\max}} \qquad \text{Eq 5-1}$$

There are two widespread ways of measuring modal analysis is that using an accelerometer or a laser vibrometer. Using an accelerometer, you get acceleration data over time. And using a vibrometer gives velocity data over time. In order to calculate the frequency response function (FRF) the fast Fourier transform (FFT) is used, in which it is possible to transform the data over time into the frequency domain. The frequency response function can be understood as the ratio between the output response of the structure and the applied load that caused the response. Due to the use of the FFT, the

FRF has both real and imaginary components. Phase and magnitude components can also be obtained and provide useful information for obtaining the dynamic parameters [221].

By detecting the peaks in the magnitude plot of FRF one can obtain the natural frequencies. These peaks are confirmed as resonant frequencies ω_r by observing a 180° variation in the phase plot at the same frequency value as the peak. The local maxima are used to determine two points ('a' and 'b') called the half power points, obtained by dividing the response value by √2. After obtaining the half power points, the loss factor η_r of this particular mode can be estimated by Eq 5-2 [222].

$$\eta_r = \frac{\omega_a^2 - \omega_b^2}{2\omega_r^2} \qquad \text{Eq 5-2}$$

In a multiple degrees of freedom system, the method called Least-squares time is commonly used [223]. In which it consists of using a curve-fitting algorithm to obtain the modal parameters, Eq 5-3[x(t)] describes the free vibration response of a system with multiple degrees of freedom.

$$x(t) = \sum_{i=1}^{N} e^{-n_i t}.\left[a_i.\sin(\omega_{di}t) + b_i.\cos(\omega_{di}t)\right] \qquad \text{Eq 5-3}$$

The coefficients a_i and b_i describe the vibration amplitude, ω_{di} represents the damped natural frequency and η_i refers to the damping ratio. By fitting the experimentally obtained response by Eq 5-3[x(t)] it is possible to identify all the modal parameters [219].

In this study, the vibration reposts were obtained using a vibrometer to acquire the data and velocity over time. In this way the FRF was processed by an FFT. To obtain the structural modal response results, the natural frequencies were extracted considering the peak-to-peak method and the structural damping value (loss factor), extracted by the model curve fitting method.

5.2.2 Artificial Neural Networks

Artificial neural networks (ANN) theory was first published in 1940 by McCulloch and Pitts, and has since been developed and has begun to acquire importance as technology advances [224]. ANN is designed in about the same way that the human brain is, with simple circuits connected by artificial neurons. The synapses formed by the connections formed between neurons are responsible for conveying impulses and distributing information, and are commonly used in learning algorithms [225]. The ANN approach has the capability of learning from data set training experience. After the training, the ANN should be able to detect output replies based on unknown input data, making it a useful tool for a variety of applications [226].

The ANN approach has the capability of learning from data set training experience. After the training, the ANN should be able to detect output replies based on unknown input data, making it a useful tool for a variety of applications [227, 228]. Weights describe the inhibitory or excitatory action of neural connections, which might be negative or positive depending on whether the connections are inhibitory or excitatory. The weight of the relevant connection (X_i x W_i) is multiplied by the value (intensity) of the received signal to compute the influence of a signal received from another neuron. The total of all X_i x W_i connections' values is added together, and the result is sent to the activation function [229]. The activation function describes how a node or nodes in a network layer convert the weighted sum of the input into an output [229].

The interaction between data and an ANN is governed by learning paradigms. Unsupervised learning and supervised learning are the two basic types of learning paradigms [230]. In supervised learning, the network is given instances and the response is compared to the desired answer. The error signal, which is the difference between the two responses, is used to change the synaptic weights in the network. This process is repeated until the network responds statistically adequately. Because the method in unsupervised learning does not strive to know the intended outputs, it does not use instances of input and output to be learned by the network. The self-organization of the ANN [229].

In addition, neural networks are classified according to how they propagate information, which can be either a feed-forward or a back-propagation network. Information flows in just one direction in feed-forward networks; that is, it flows from the input layer to the output layer in only one direction [231]. Information can travel back and forth between layers in backpropagation networks [232]. For an adequate ANN configuration, you should design a model that is not too stiff to not correctly describe the data, but also not too flexible to model the data's noise.

All perceptron-solvable issues can be resolved with just one hidden layer, however there are situations when using two or three hidden layers is more effective. The network's predictions for the outside environment are then produced by the neurons in the output layer [233]. A typical neural network is depicted in Figure 5-1 with input, output, sum function, and sigmoid activation function. The output of the linked neuron is multiplied by the synaptic strength of the connection between them to determine the input to a neuron from another neuron. Using Eq 5-4, the weighted sums of the input components $(net)_j$ are computed.

$$\text{net}_j = \sum_{i=1}^{n} w_{ij} o_i + b \qquad \text{Eq 5-4}$$

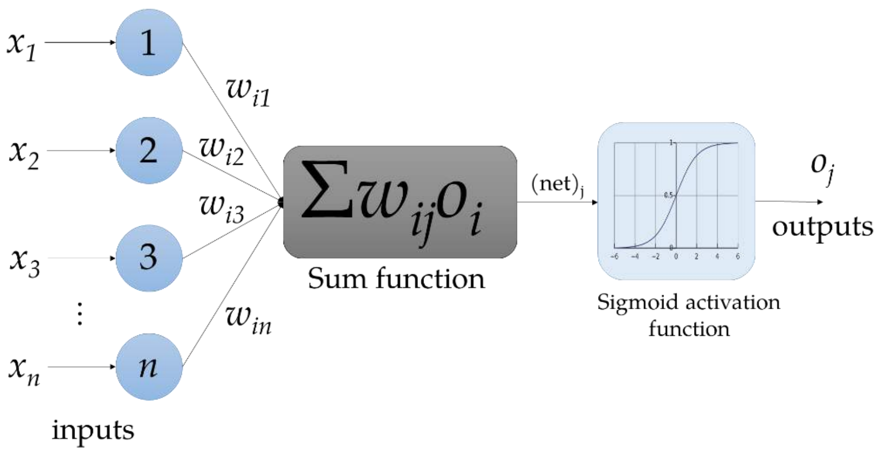

Figure 5-1: The artificial neuron model (adapted from (Sarıdemir, 2009).

Where $(net)_j$ is the weighted sum of the j_{th} neuron for the input received from the preceding layer with n neurons, w_{ij} is the weight between the j_{th} neuron in the preceding layer, oi is the output of the ith neuron in the preceding layer. The network input $(net)_j$ and the neuron's output signal (o_j in Figure 5-1) are connected by a transformation function known as the activation function. Ramp, sigmoid, and Gaussian functions are the most often used activation functions. In general, for multilayer receptive models as the activation function $(f(net)_j)$ sigmoid function is used. The output of the j_{th} neuron o_j is calculated by using Equation 4 with a sigmoid function as shown in Eq 5-5.

$$o_j = f\left(\text{net}_j\right) = \frac{1}{1 + e^{-\alpha\,\text{net}_j}} \qquad \text{Eq 5-5}$$

Where a is constant that regulates how steeply the semi-linear region slopes. With the exception of the input layer, every layer has the sigmoid nonlinearity active. The outputs (0,1) of the sigmoid function described by Eq 5-5.

A network of neurons dispersed over three or more layers makes up the feedforward multilayer perceptron network (ANN) (see Figure 5-2). Although there are no connections between neurons in the same layer, every neuron in each of these layers is entirely connected to every neuron in the layer above it and the one below it. The input layer, also known as the ANN input parameters, is the initial layer and has the same number of neurons as inputs. The last is the output layer i.e. the ‘‘results’’ of the ANN, with the same number of neurons as problem outputs. The layer or layers between these two are known as the hidden layers. The number of hidden layers and the number of neurons in each hidden layer depend on the problem under study and cannot be known beforehand [234].

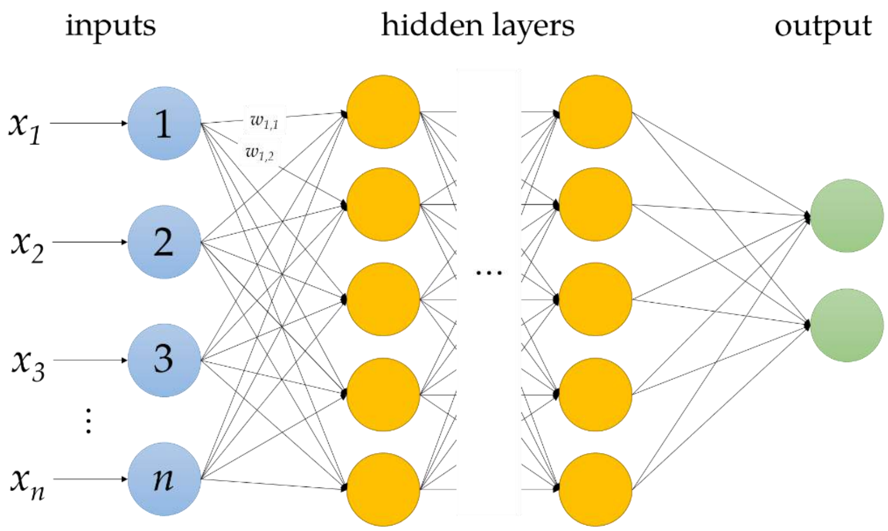

Figure 5-2: Feedforward Neural Network.

5.3 Experimental methodology

5.3.1 General Full Factorial Design

The systematic method called Design of experiments (DOE) is used to determine the relationship between factors that affect a process and its results [235]. Usually, the DOE method can be divided into full factorial design (FFD) and fractional factorial design also known as Taguchi Experimental Design (TED). In FFD design, all combinations of the parameter levels are tested in order to analyze the results. The so-called General full factorial design (GFFD) is a variation of FFD, which allows running more than two levels per variable [236]. The variables and their ranges are given in Table 5-1. In the GFFD setup there are 3 levels with 4 control factors S/C, CS, FES, and PU. The tests were performed in triplicate for each mixture, the software used for the analyses was Minitab version 2018.

Table 5-1: Input factors and their levels.

Control variables	Abbreviation	Symbol	Level			
Sand by cement	S/C	X_1	1		3	
Coarse Sand Ratio	SC	X_2	20%		50%	
Foundry Exaust Sand	FES	X_3	10%		40%	
Polyurethane	PU	X_4	0%	10%	30%	60%

The Table 5-2 shows the characterization of the materials used to prepare the mortar.

Table 5-2: Physical properties of fine and coarse aggregates

Properties	Cement	Lime	NS	PU	FES
Density (kg/m³)	3140	1600	2650	660	2700
Specific surface (m²/kg)	330	-	5	20	29.4
Initial setting time (min)	120	-	-	-	-
Final setting time (min)	500	-	-	-	-
Water absorption (%)	-	-	0.95	0.05	0.75
Unit Weight (kg/m³)	1440	1120	1350	220	1450

In Table 5-1, S/C represents two families of 1:1 to 1:3 (read, one parts binder to three parts sand) mixtures, with the binder consisting of cement plus hydrated lime in the ratio of 4:1, i.e., four parts cement and one part lime. The CS denotes the percentage of coarse sand replacing fine sand from 20% to 50%, where the coarse sand has a granulometry ranging from 1.2mm to 4.8mm and the fine sand from 0.75mm to 1.2mm; the mass of FES replaces NS from 10% to 40%; the mass of PU replaces NS from 0%,10%, 30% to 60%. The water to cement ratio (w/c) in the 1:1 mix was equal to 0.45 and for the 1:3 mixtures it was 0.60. The superplasticizer content was 1.0 of the cement weight for all mixtures. With these proportions were obtained spreads in the fresh state of 260 ± 25mm recommended by the standard [165] for coating mortar use that was taken as the limit for control of the study.

With these proportions, measures of fresh state spreading of 260 ± 50 mm were obtained, close to that recommended by the standard [165] for the use of coating mortar, which was taken as the limit for the study control. A standard deviation of ±50 mm was allowed because, some combinations of proportions obtained facilitated spreading and others hindered by the incorporation of FES and PU waste. As the focus of this study is only on dynamic properties, we fixed this range for study so that we could compare the groups without changing w/b ratio and/or $\%S_p$.

By means of the experimental arrangement chosen in the GFFD with 3 variables of 2 levels and 1 variable of 4 levels, the number of 32 mixtures of MPUFS was reached. For the mixtures, three replicates were performed in order to increase the reliability of the results. Thus, a total of 96 samples of MPUFS were tested for the complete development of the GFFD and, consequently, the generation of the ANN. MATLAB software version 2019 was used to run and analyze the ANN.

The Table 5-3 shows all mixtures with the decoded units and consumption of all materials per mixture. The notations used in this Table 5-3 (M_1, M_2, … M_n) represent the mixtures and their respective number in the numbering sequence. Although the experiments appear in the Table 5-3 in ascending order, they were performed in a random order in the laboratory to avoid possible biases. The mixtures M_{R1}, M_{R2}, M_{R3}, M_{R4} represent the reference mixtures without incorporation of FES and PU residues.

(Space intentionally left)

Table 5-3: Uncoded units and consumption of materials by m³

Mixtures code	SC	S/C	FES	PU	Cement(g)	Lime(g)	NS(g)	FES(g)	PU(g)	Sp(g)	w/c
M_1	20%	1	10%	0%	786.62	196.66	707.96	78.7	0.00	7.87	0.45
M_2	20%	1	10%	10%	786.62	196.66	688.16	78.7	19.80	7.87	0.45
M_3	20%	1	10%	30%	786.62	196.66	648.56	78.7	59.40	7.87	0.45
M_4	20%	1	10%	60%	786.62	196.66	589.17	78.7	118.80	7.87	0.45
M_5	20%	1	40%	0%	786.62	196.66	471.97	314.6	0.00	7.87	0.45
M_6	20%	1	40%	10%	786.62	196.66	452.18	314.6	19.80	7.87	0.45
M7	20%	1	40%	30%	786.62	196.66	412.58	314.6	59.40	7.87	0.45
M_8	20%	1	40%	60%	786.62	196.66	353.18	314.6	118.80	7.87	0.45
M_9	20%	3	10%	0%	455.70	113.92	1321.52	45.6	0.00	4.56	0.60
M_{10}	20%	3	10%	10%	455.70	113.92	1287.11	45.6	34.41	4.56	0.60
M_{11}	20%	3	10%	30%	455.70	113.92	1218.30	45.6	103.23	4.56	0.60
M_{12}	20%	3	10%	60%	455.70	113.92	1115.07	45.6	206.46	4.56	0.60
M_{13}	20%	3	40%	0%	455.70	113.92	1184.81	182.3	0.00	4.56	0.60
M_{14}	20%	3	40%	10%	455.70	113.92	1150.41	182.3	34.41	4.56	0.60
M_{15}	20%	3	40%	30%	455.70	113.92	1081.59	182.3	103.23	4.56	0.60
M_{16}	20%	3	40%	60%	455.70	113.92	978.36	182.3	206.46	4.56	0.60
M_{17}	50%	1	10%	0%	786.62	196.66	707.96	78.7	0.00	7.87	0.45
M_{18}	50%	1	10%	10%	786.62	196.66	688.16	78.7	19.80	7.87	0.45
M_{19}	50%	1	10%	30%	786.62	196.66	648.56	78.7	59.40	7.87	0.45
M_{20}	50%	1	10%	60%	786.62	196.66	589.17	78.7	118.80	7.87	0.45
M_{21}	50%	1	40%	0%	786.62	196.66	471.97	314.6	0.00	7.87	0.45
M_{22}	50%	1	40%	10%	786.62	196.66	452.18	314.6	19.80	7.87	0.45
M_{23}	50%	1	40%	30%	786.62	196.66	412.58	314.6	59.40	7.87	0.45
M_{24}	50%	1	40%	60%	786.62	196.66	353.18	314.6	118.80	7.87	0.45
M_{25}	50%	3	10%	0%	455.70	113.92	1321.52	45.6	0.00	4.56	0.60
M_{26}	50%	3	10%	10%	455.70	113.92	1287.11	45.6	34.41	4.56	0.60
M_{27}	50%	3	10%	30%	455.70	113.92	1218.30	45.6	103.23	4.56	0.60
M_{28}	50%	3	10%	60%	455.70	113.92	1115.07	45.6	206.46	4.56	0.60
M_{29}	50%	3	40%	0%	455.70	113.92	1184.81	182.3	0.00	4.56	0.60
M_{30}	50%	3	40%	10%	455.70	113.92	1150.41	182.3	34.41	4.56	0.60
M_{31}	50%	3	40%	30%	455.70	113.92	1081.59	182.3	103.23	4.56	0.60
M_{32}	50%	3	40%	60%	455.70	113.92	978.36	182.3	206.46	4.56	0.60
M_{R1}	20%	1	0%	0%	786.62	196.66	786.62	0.00	0.00	7.87	0.45
M_{R2}	20%	3	0%	0%	455.70	113.92	1367.09	0.00	0.00	4.56	0.60
M_{R3}	50%	1	0%	0%	786.62	196.66	786.62	0.00	0.00	7.87	0.45
M_{R4}	50%	3	0%	0%	455.70	113.92	1367.09	0.00	0.00	4.56	0.60

5.3.2 Manufacturing and materials

The materials used in this study were: cement type CP II-E-32 [154], hydrated lime ABNT NBR 7175 (2003) [155] as binders, natural sand (NS), foundry exhaust sand (FES), polyurethane powder (PU) as aggregate.

The FES has a predominant composition of SiO_2, Fe_2O_3 which was obtained by X-ray fluorescence analysis. The PU has a predominant composition of C, H, O, N, respectively which was obtained by infrared spectroscopy analysis. The Figure 4-1 represents the particle distribution of the binder (cement and lime), obtained by the laser ray diffraction method and aggregates (NS, FES, PU) obtained by sieving on the normal sieve series [156, 157]. Potable water was used for molding and curing the samples. Furthermore, according to [158], 1.5% of water reducing admixture, 3rd generation superplasticizer based on modified synthetic carboxylated polymers with specific weight of 1.24 kg/lit) was used to increase the consistency of the MPUFS with the lowest possible (w/b) ratio.

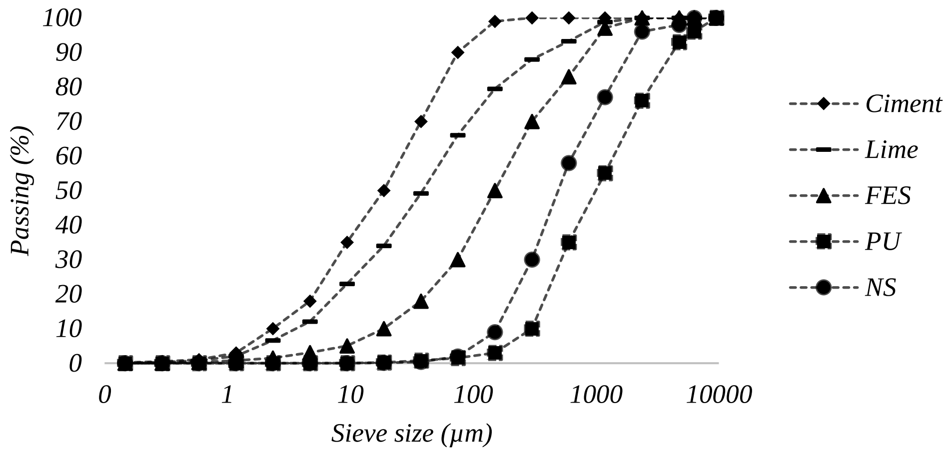

Figure 5-3: Particle size distribution.

In the current study, FES and PU were used as fine aggregate and replaced the NS in mass in different percentages. The PU used comes from the recycling of household appliances, more specifically from domestic refrigerators; it was obtained through a donation from the company Industria Fox®. The recycling process used by Industria Fox® consists first of filtering the refrigeration gases Chlorofluorocarbon (CFC) that contribute to the destruction of the ozone layer, and persistent organic contaminants such as mercury present in the refrigerator compressor. Such gases are treated by a chemical process that transforms the gas into an acid solution, so that it may be used later by the chemical industry. The leftovers of the device are shredded and the remains of plastic, iron, aluminum and any other materials that can be reused are separated and sent to recycling companies and cooperatives [159].

The foundry waste industry produces countless by-products, from used or residual foundry sand, slag, ash, refractories, coagulants, dust from the exhaust system (filters), scrap, vapors and residual kiln liquids [10, 160]. In the current study, the FES was obtained through donation from the company Mahle Metal Leve AS®, Itajubá. The FES is a type of foundry sand waste that is generated by mixing sand, bentonite and charcoal in the manufacturing process of green sand molds for casting metal parts. During the release of the parts by vibratory transport, the very fine sand particles that remain in suspension are filtered through bag filters [161]. It is necessary to remove FES from the casting process, because due to its powdery size it impairs the permeability of the molds and makes it difficult for the process gases to escape. With this, bubbles and harmful voids can be formed in the castings [162].

First, the dry materials, cement, aggregates were mixed with two thirds of the total volume of water and then mixed for 2 min. Then the remaining water and the superplasticizer were slowly added and mixed for another 4 min. Next, consistency index test was performed on MPUFS in the fresh state according to the standard [165]. Finally, the fresh MPUFS was placed in the molds for the physical and mechanical tests, respectively. The samples remained in an environment with relative humidity higher than 90% for 24h and then were submerged in drinking water in a curing tank with saturated lime water at 21 ± 3°C.

The first two tests that were performed were the voids content and absorption by immersion, according to the prescriptions of [116], for mortar with 28 days of age, using specimens with 50 mm in diameter and 100 mm in height. Three samples were molded for each mortar mixture developed.

The mortars tested in the spreading test are shown in Figure 5-4. All mortars had a slump range of 250±50mm. The mixtures in the Figure 5-4 were carefully chosen among the various mixtures produced in this study, because they represent well how some visual changes occurred in the fresh state. It is worth noting that the study does not intend to analyze the fresh state characteristics of the mortar, but the authors found it worthwhile to represent the change of physical aspect that occurred in the mixtures during the incorporation of waste and the change of some parameters such as S/C and w/c content.

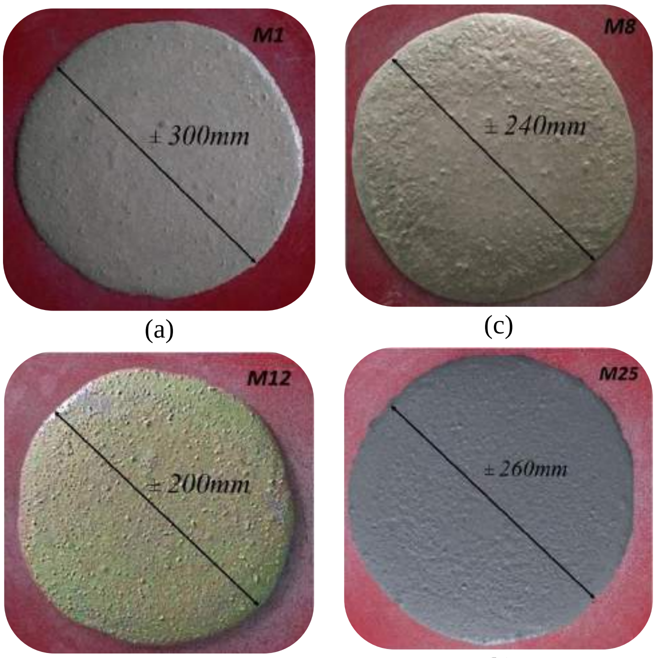

Figure 5-4: Spreading test of the MPUFES samples. (a) sample M_1, (b) sample M_8, (c) sample M_{12}, (d) sample M_{25}.

5.4 Experimental Setup

The MPUFS samples were evaluated for forced vibration tests in which the external force was produced by a shaker (small hammer). The equipment used in the tests is shown in Figure 5-5. For the experimental tests were performed considering a free, or unsupported, configuration. For this, metallic cables were used to support the sample during the execution of the shaker impact. The Figure 5-5 represents the test setup, in which is formed by a sensor, a vibrator, a data acquisition and signal analysis system, note a metallic support was used for the specimens to reach the free vibration or unsupported condition. The instruments used in the experiment include LabVIEW programming, Data Acquisition board (DAQ), laser sensor and shaker. A DAQ plate used was a photon model from Brüel&Kjær. A laser vibrometer, model VQ-500-D from Ometron, was used for measuring the vibration displacement at a targeted point. For the free vibration analysis, an impact hammer, Brüel & Kjær, was used in the modal experimental test. The vibration shaker, model 4808 from Brüel & Kjaer, was triggered by the voltage signal from the power amplifier, model 2712 from Brüel & Kjaer. The RT Pro Photon program was used to process all the signals obtained.

Cementitious products in general have low reflective capacity, so for signal acquisition it was necessary to position a plastic tape to assist in signal capture. As the tape has very low thickness and mass compared to the MPUFS samples, it was considered that its presence did not affect the collection of vibration results. It is worth noting that the laser vibrometer for this type of experiment can be considered more suitable than the accelerometer, since the laser is not influenced by the total mass of the system, thus differences in mass of the samples do not affect the vibration results.

In the present study, the modal properties of the different specimens manufactured designs are carried out by acquiring vibration signals with the help of a laser vibrometer (sensitivity 8000 m/s/mV). The sensor is pointed at the surface of the specimen (with the aid of a reflective tape). The output of the vibrometer is fed into the Brüel & Kjaer Photon+ DAQ through a USB chassis where the analog vibration signals are conditioned and converted into digital form. The impulse force is applied by using an impact hammer (sensitivity 21.08 $m/s^2/mV$). In order to ensure Shannon's sampling theorem, the sampling frequency was set to 1500Hz.

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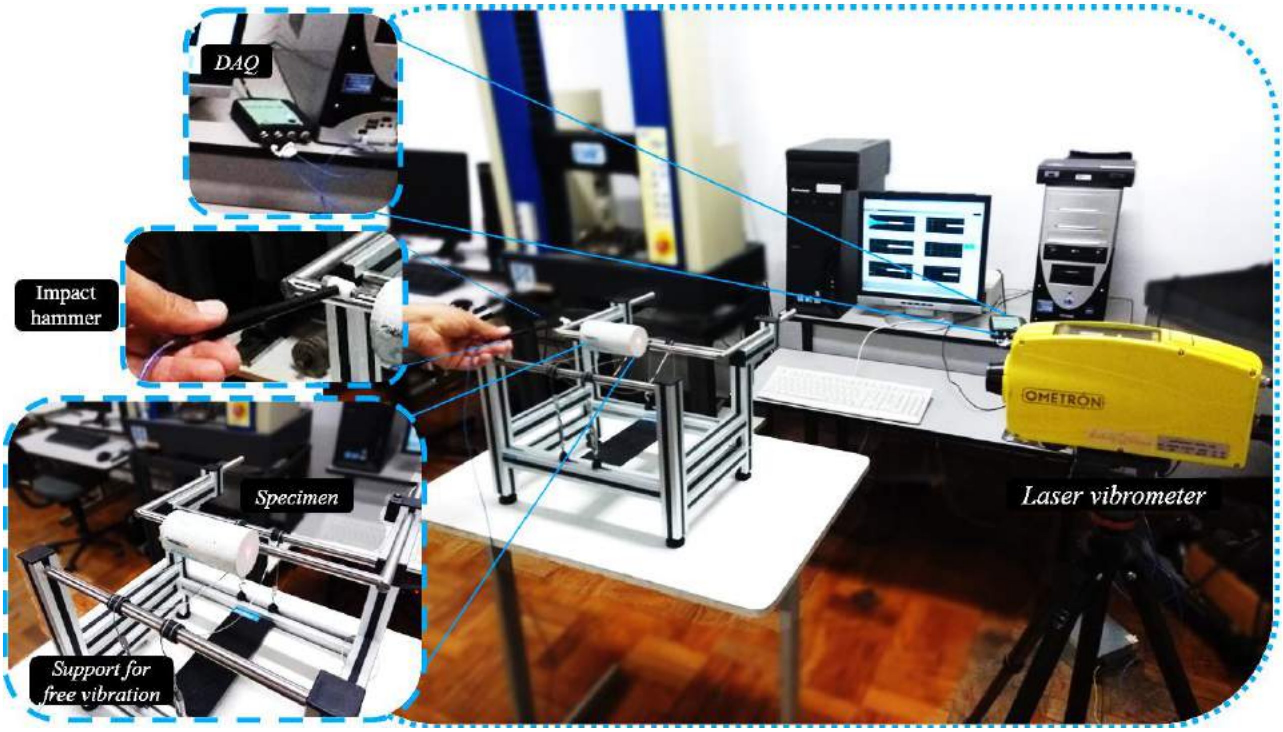

Figure 5-5: General experimental setup and details for free vibration.

The experimental steps of this study have been summarized in Figure 5-6. It can be seen that for the production of the MPUFS it was necessary to perform important steps. The first of them is the execution of the General Full Factorial Design of experiments (GFFD), in which a set of variables was worked on at different levels, which can be seen in Table 5-1. Then the modal tests were performed and after the analysis of variance, the modeling of the mixing and optimization designs was performed in two ways. One was to use the quadratic model obtained in the RSM-BB configuration used. The other was the use of Artificial Neural Network to arrive at a Modal Properties prediction that will generate a modeling of the mixing projects. This way it will be possible to compare the feasibility of both procedures.

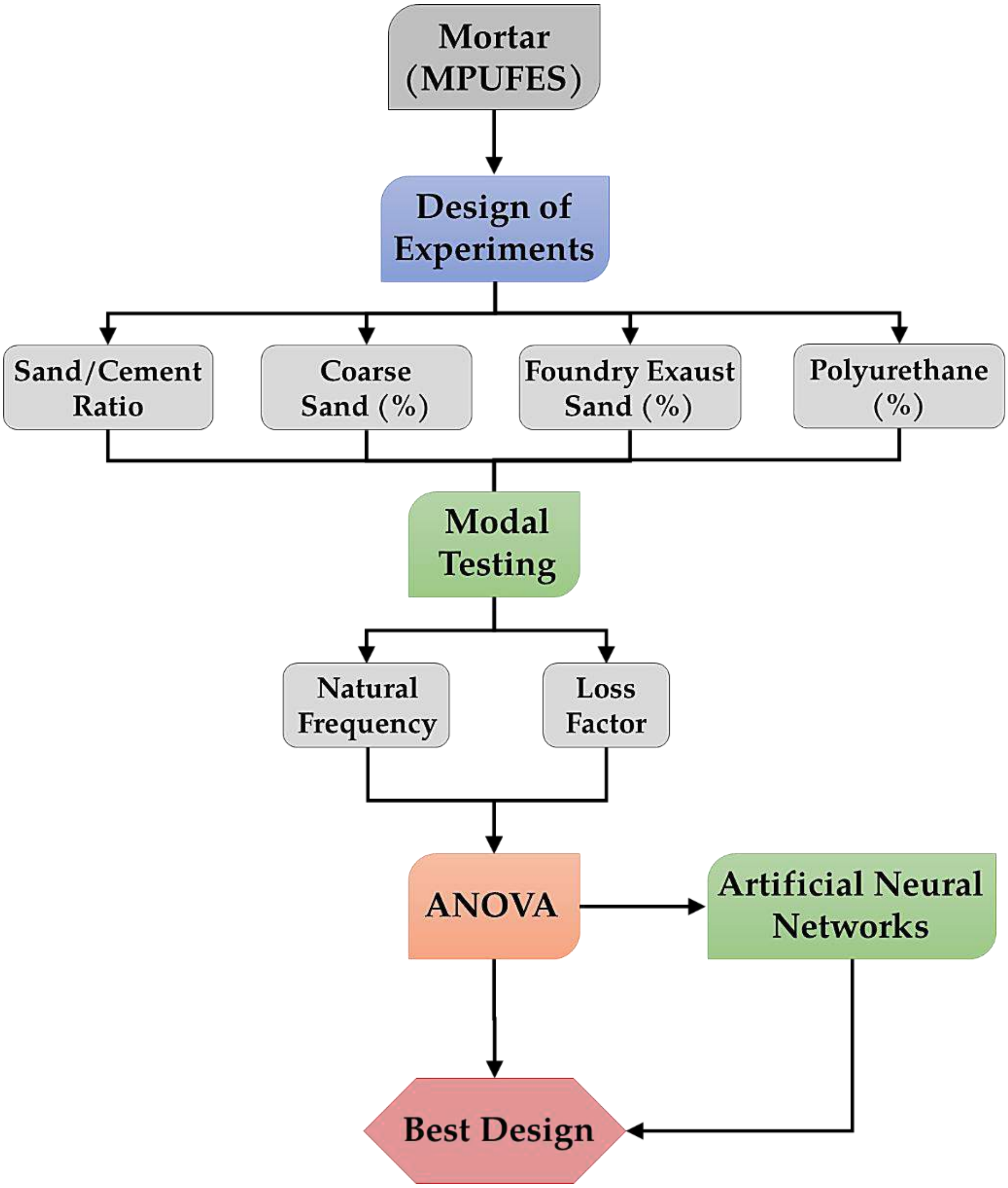

Figure 5-6: General flowchart for the full development of the MPUFS study.

5.5 Experimental results

In this section, the results obtained by the proposed method are discussed. The first analysis was a free-vibration and forced-vibration analysis, which allowed the responses of residual mortars to be compared with a reference. Statistical results are then evaluated using the RSM and ANOVA methods. Finally, the results of the ANN are presented.

5.5.1 Modal Results

The first experimental test was for free vibrations using the configuration described in Figure 5-5. Free vibration is the response of a system to an initial input, allowing it to vibrate freely. Through it is possible to analyze the natural frequencies and their amplitudes, as well as the damping or loss factor of the system.

The Figure 5-7 shows the main response of the free vibration time histories of MPUFES composites with all the different design factors tested at the same initial vibration amplitude. The evaluation of the responses obtained in Figure 5-7 can be done based on the comparison of the tested material to the adopted benchmark. The damping phenomenon is more pronounced in the samples from M_{13} to M_{15} and M_{30} to M_{32}, in these samples the material presents fast absorption of the oscillation amplitude. In contrast, the samples from the groups M_1 to M_4 and M_{17} to M_{20} present low absorption of the oscillation amplitude.

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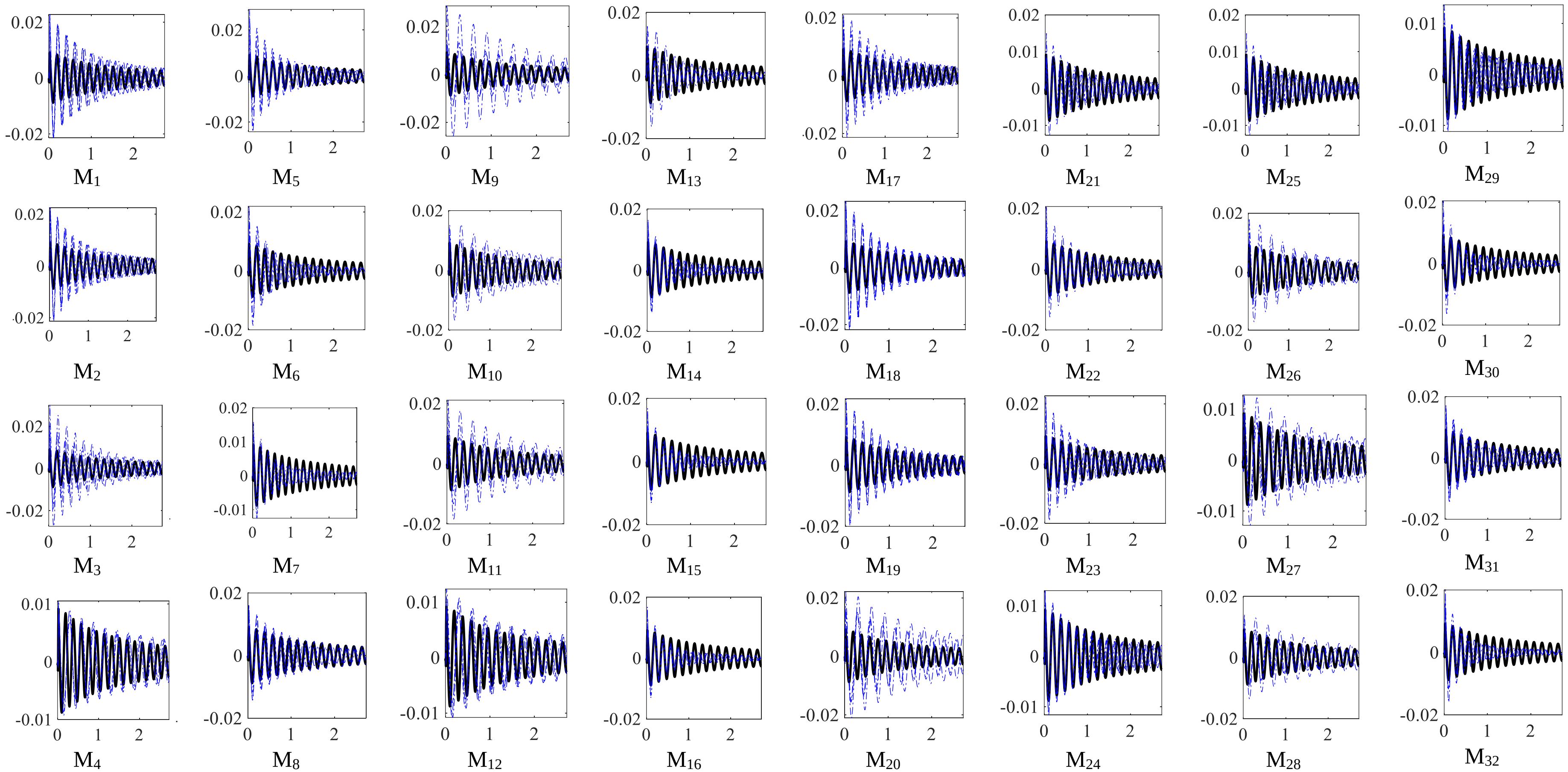

Figure 5-7: Velocity time response for considering the different design factors (legend: ▬ specimens and ▬ reference).

Using the Fast Fourier Transform, the damping result expressed in velocity by time is converted into the frequency domain. With the natural frequency of the tested material as a parameter, the Frequency Response Function (FRF), which will offer the initial vibration mode, is generated in this way. In Figure 5-8 it is possible to observe that in general the natural frequency of all tested mortars did not change significantly in the first vibration mode or resonant frequency in comparison to the reference sample. The frequency found is close to 5 Hz.

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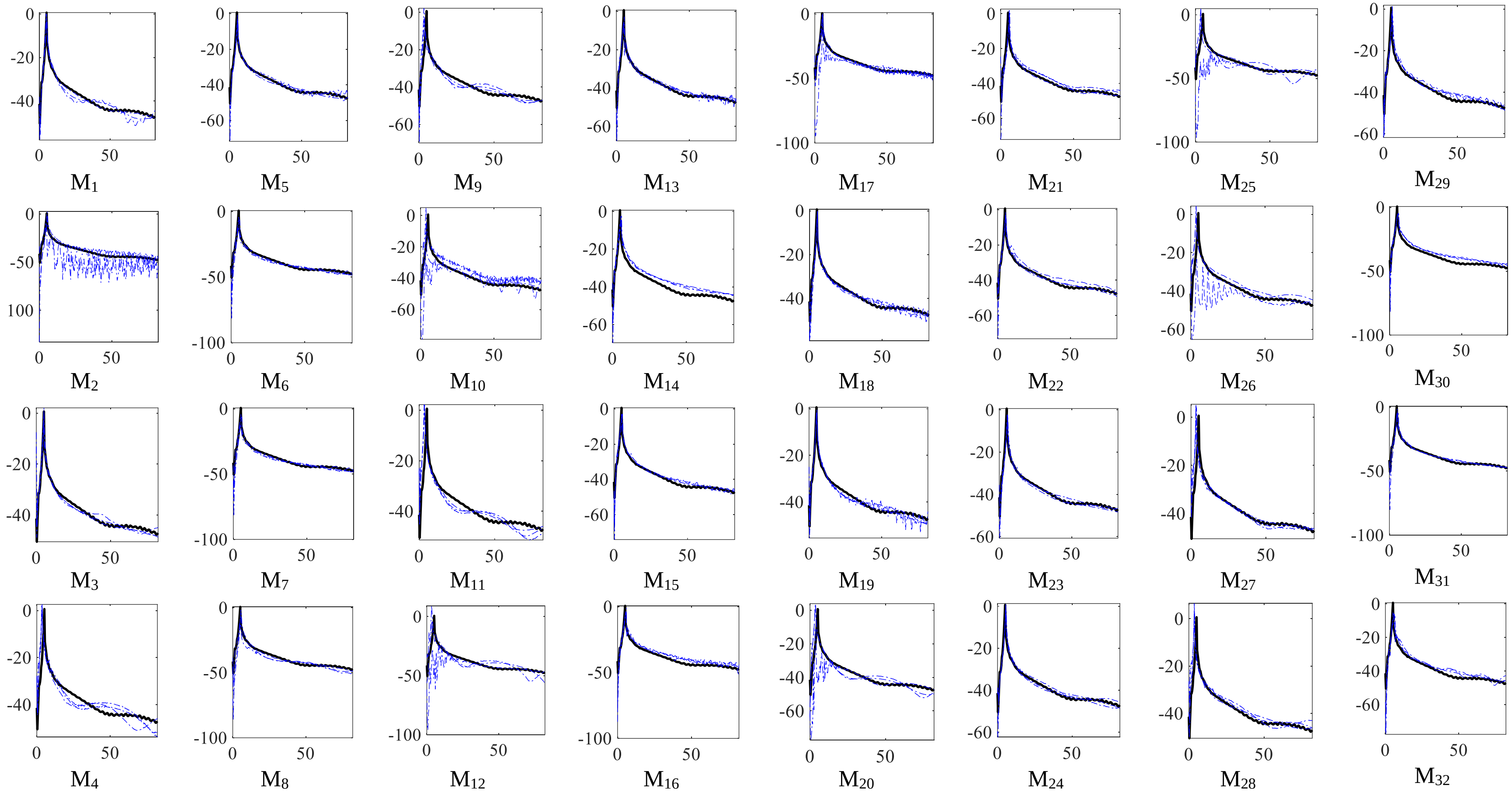

Figure 5-8: Frequency response function for considering the different design factors (legend: ▬ specimens and ▬ reference).

5.5.2 Analysis of Variance

The main objective of this research is to dynamically characterize mortars with PU and FES waste incorporation, named as MPUFES. The results were conducted by means of an experimental planning using an RSM, the parameterization was based on four manufacturing factors and two responses, (see Table 1). The factors were coarse sand content (SC), sand to cement ratio (S/C), replacement content of natural sand with foundry exhaust sand (FES), replacement content of natural sand with polyurethane (PU). The responses were natural frequency and loss factor. Figure 5-9 and Figure 5-10 represent the results obtained by averaging an analysis of variance (ANOVA) of the responses as a function of the manufacturing factors.

To analyze the statistical results the modal responses were normalized by percentage change analysis, as shown in Eq 5-6 and Eq 5-7. The Table 5-4 summarizes the overall results of this study with the experimental setup and responses.

$$\Delta\omega = (1 - \frac{\omega}{\omega_{ref}}) \times 100 \qquad \text{Eq 5-6}$$

$$\Delta\eta = (1 - \frac{\eta}{\eta_{ref}}) \times 100 \qquad \text{Eq 5-7}$$

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Table 5-4: Vibrations responses for all the different manufacturing design of MPUFES

Variables					Experimental Responses	
Exp.	Coarse Sand (%)	Sand/ Cement	Foundry Ex. Sand (%)	PU (%)	ω_n (Hz)	η (%)
M_1	0.2	1	0.10	0.00	5.127	3.280
M_2	0.2	1	0.10	0.10	5.493	7.700
M_3	0.2	1	0.10	0.30	3.296	4.780
M_4	0.2	1	0.10	0.60	5.493	0.070
M_5	0.2	1	0.40	0.00	5.127	3.040
M_6	0.2	1	0.40	0.10	5.859	3.800
M_7	0.2	1	0.40	0.30	9.888	5.000
M_8	0.2	1	0.40	0.60	6.226	4.000
M_9	0.2	3	0.10	0.00	4.761	6.180
M_{10}	0.2	3	0.10	0.10	6.226	11.580
M_{11}	0.2	3	0.10	0.30	2.930	1.020
M_{12}	0.2	3	0.10	0.60	5.493	10.360
M_{13}	0.2	3	0.40	0.00	5.493	2.920
M_{14}	0.2	3	0.40	0.10	6.226	16.700
M_{15}	0.2	3	0.40	0.30	4.028	1.010
M_{16}	0.2	3	0.40	0.60	5.493	5.500
M_{17}	0.5	1	0.10	0.00	5.127	3.280
M_{18}	0.5	1	0.10	0.10	5.127	7.940
M_{19}	0.5	1	0.10	0.30	3.296	3.390
M_{20}	0.5	1	0.10	0.60	5.859	5.220
M_{21}	0.5	1	0.40	0.00	5.127	3.210
M_{22}	0.5	1	0.40	0.10	5.493	4.880
M_{23}	0.5	1	0.40	0.30	3.662	1.550
M_{24}	0.5	1	0.40	0.60	5.493	5.560
M_{25}	0.5	3	0.10	0.00	3.296	4.040
M_{26}	0.5	3	0.10	0.10	5.859	14.780
M_{27}	0.5	3	0.10	0.30	8.057	5.000
M_{28}	0.5	3	0.10	0.60	5.127	8.000
M_{29}	0.5	3	0.40	0.00	3.662	2.750
M_{30}	0.5	3	0.40	0.10	5.127	13.460
M_{31}	0.5	3	0.40	0.30	3.662	2.300
M_{32}	0.5	3	0.40	0.60	5.859	7.680
M_{R1}	0.2	1	0.00	0.00	5.133	5.090
M_{R2}	0.2	3	0.00	0.00	5.110	5.326
M_{R3}	0.5	1	0.00	0.00	5.055	5.800
M_{R4}	0.5	3	0.00	0.00	5.125	5.051

The Figure 5-9 shows the main effects plot results for the first natural frequency, where for larger sizes, we have smaller values of the natural frequency, while for density, it occurs in the opposite way. A higher value of natural frequency is discovered for greater values. The behavior of the loss factor's primary effects is depicted in Figure 5-10. A more in-depth study is challenging in this situation since the size variable does not exhibit a clearly defined pattern. However, for density, we have that the loss factor is higher for higher densities.

The Figure 5-9 displays the major impact plot results for the first natural frequency, where lower natural frequency values are observed for larger SC and S/C concentrations. Higher values of natural frequency correspond to higher FES levels. The impact of the PU contents is not well defined; there is no clear pattern within the items that are utilised.

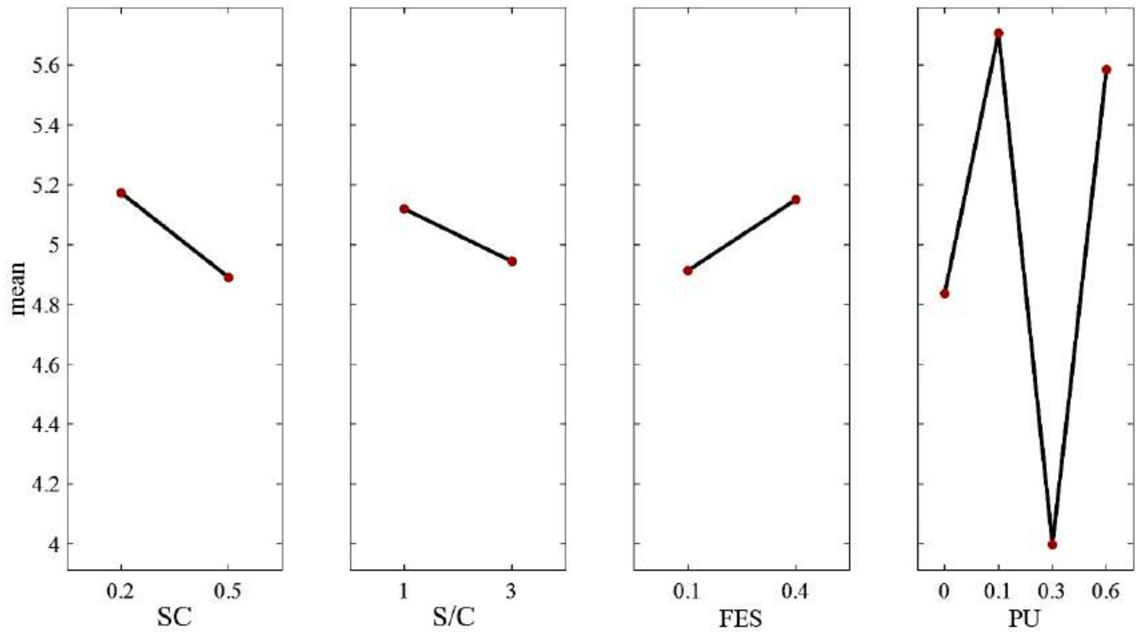

Figure 5-9: Main effect plot for the first natural frequency.

The main impacts plot results for the loss factor are displayed in Figure 5-10. Take note of how the tendencies matched those found for the first natural frequency. Naturally occurring frequency values are lower for higher SC and S/C levels. Higher values of natural frequency correspond to higher FES levels. The impact of the PU contents is not well defined; there is no clear pattern within the items that are utilized.

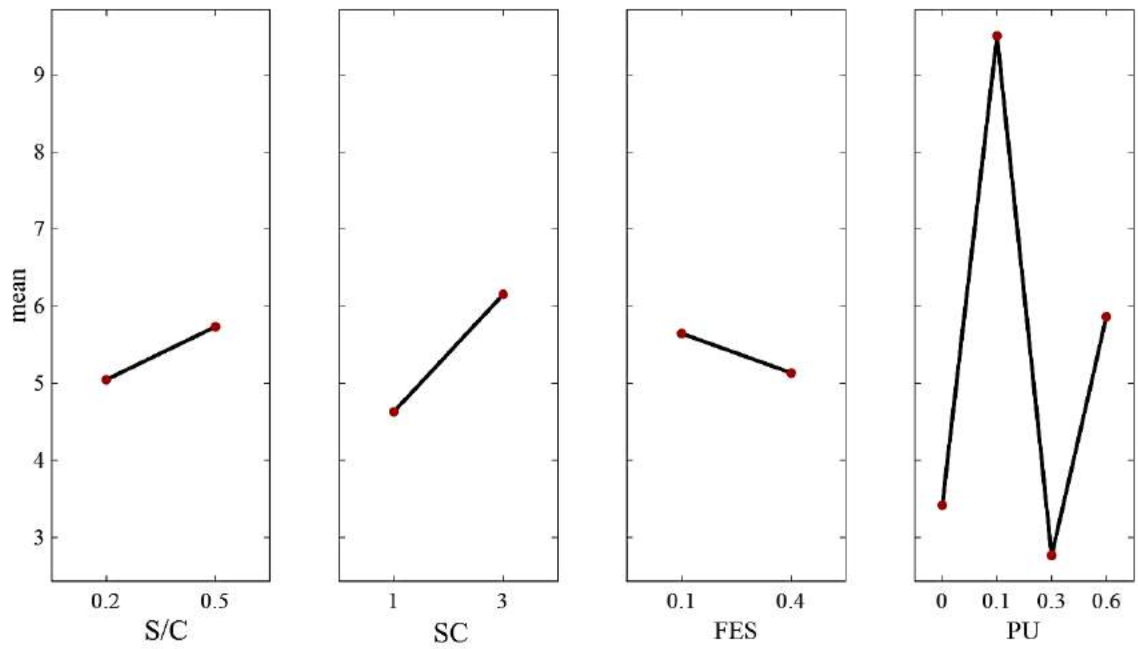

Figure 5-10: Main effect plot for plot for the loss factor.

The Interaction plot for the first natural frequency is shown in Figure 5-11 and the Interaction plot for the loss facto is shown in Figure 5-12. The initial natural frequency and loss facto proportionately are often modified by the researched variables at the various levels tested. The variables are not proportional only in a few limited circumstances. In this instance, SC interacts with PU in a percentage range of 0% to 10%.

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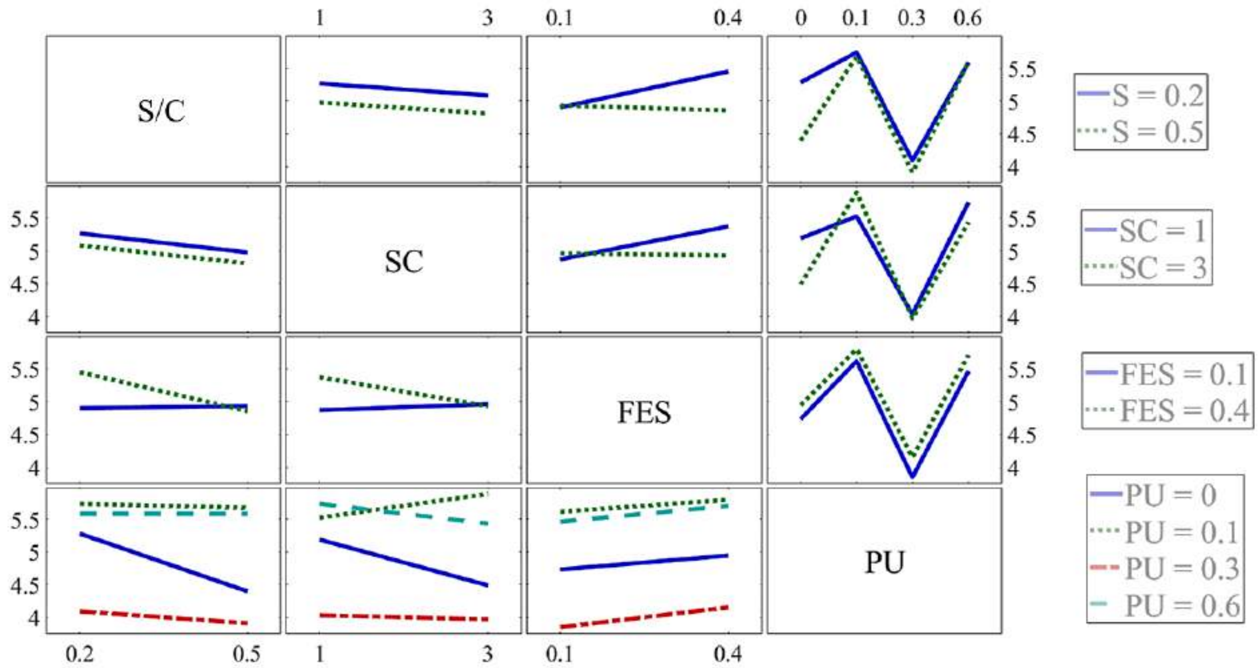

Figure 5-11: Interaction plot for the first natural frequency.

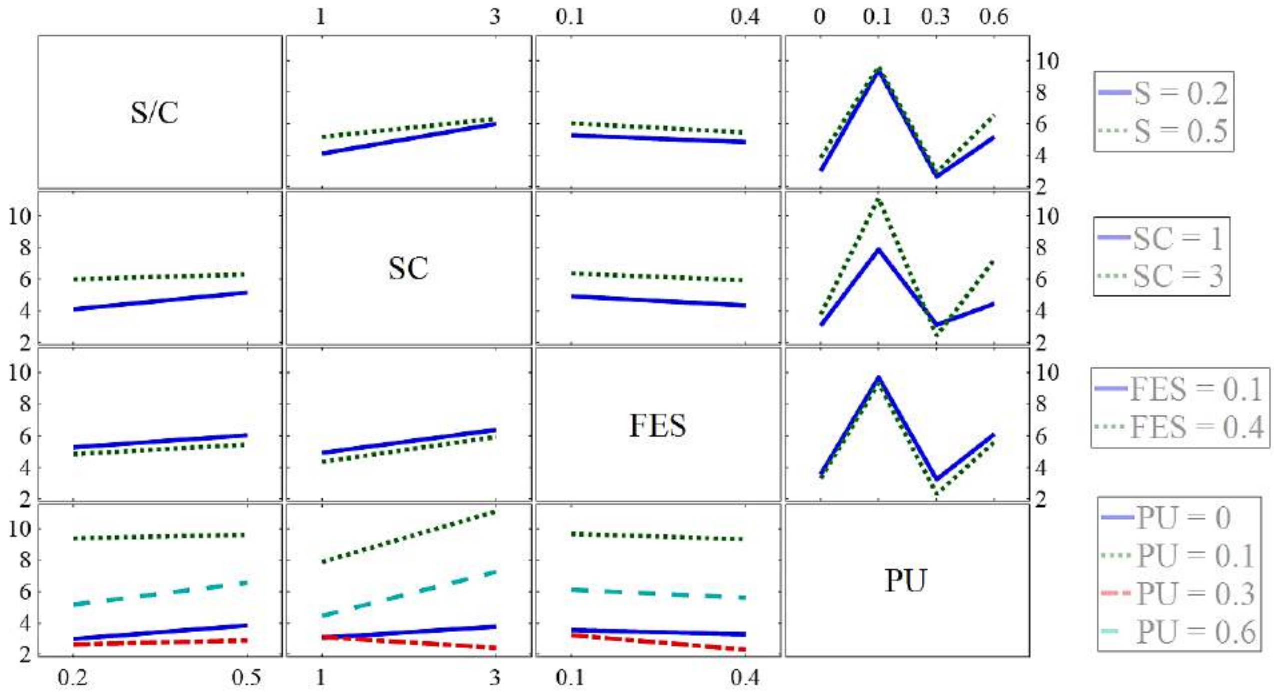

Figure 5-12: Interaction plot for the loss factor.

Integrating the analyses, Figure 5-13 displays the response surface combining the two variables, allowing for analysis of their combined effects on natural frequency, loss factor, vibration amplitude, and mass. Natural frequency, loss factor, and amplitude displayed non-linear behavior on the in-space response surfaces, however the mass displayed near-plane (linear) behavior.

In Figure 5-13 the combination of analyses represented by means of the response surface was performed. In this scenario it is possible to analyze the combination of the effects of the variables on the modal responses tested. Figure 5-13a and Figure 5-13c behave in a similar manner in which linear behavior occurs. The variables S/C and SC are linear and inversely proportional to ω_n. In Figure 5-13a, the maximum value of ω_n occurs for the lowest values of SC and S/C which represents an increase of ωn by 2.8% over the average value (reference mixture). In Figure 5-13c the pattern is the reverse. For SC and S/C increase, the N increase reaches 27.0% of the average value.

The Figure 5-13b the incorporation of PU modifies the ω_n not having a pattern within the analyzed spectrum. However, it can be seen that some points of extreme low and high absolute values occur. This is the case for the percentage of 30% PU in which the ω_n has a decrease of -27.5% in relation to the average value. In both Figure 5-13c and Figure 5-13d, the percentage of 10% PU causes an increase close to 66.0% in η, if compared to the average value (reference mixture). The sharp decrease in natural frequency and loss factor for 30% polyurethane waste and 0.1 cast exhaust area waste Figure 5-13(b) and Figure 5-13(d) as it was to be analyzed is directly related to the decrease in loss factor. A low loss factor material represents a material that is able to absorb more impacts in a shorter time interval, i.e., cushion impacts better [237].

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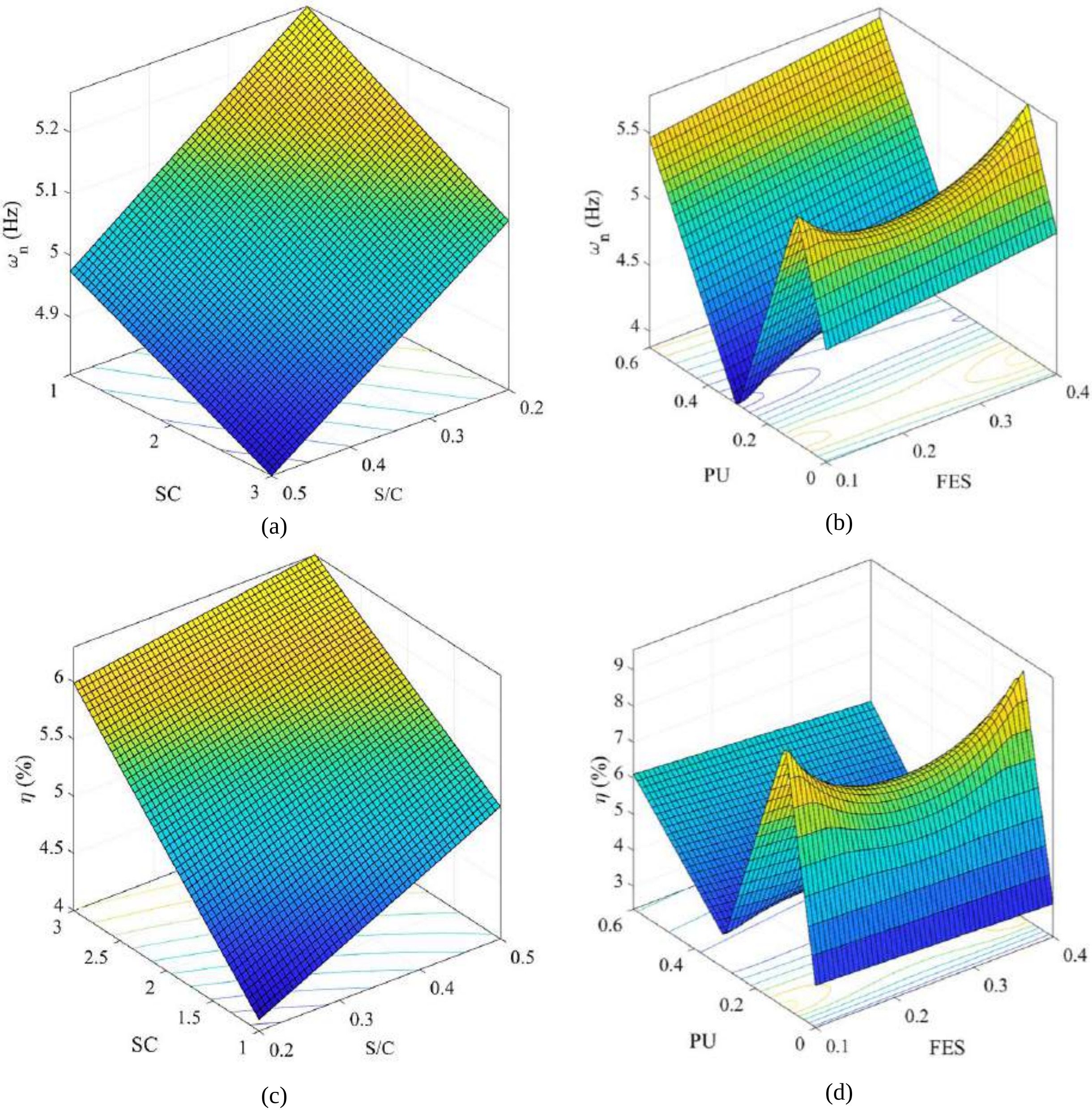

Figure 5-13: Response surfaces in the space for (a) ω_n by the variables SC x S/C; (b) ω_n by the variables PU x FES, (c) η by the variables SC x S/C e (d) η by the variables PU x FES.

5.5.3 Modal Properties Prediction using ANN

In this section, an ANN was developed to predict the modal responses regarding the modal behavior model of mortars with PU and FES waste. The input layer is composed of design parameters (4 variables) such as coarse sand content (SC), sand to cement ratio (S/C), percentage of FES and percentage of PU replacing natural sand. The output layer is composed of the modal responses of natural frequency, and loss factor or damping rate. Thus, the ANN created has one input layer with four neurons, a hidden layer with 20 neurons and an output layer with 2 neurons, as shown in the architecture of Figure 5-14.

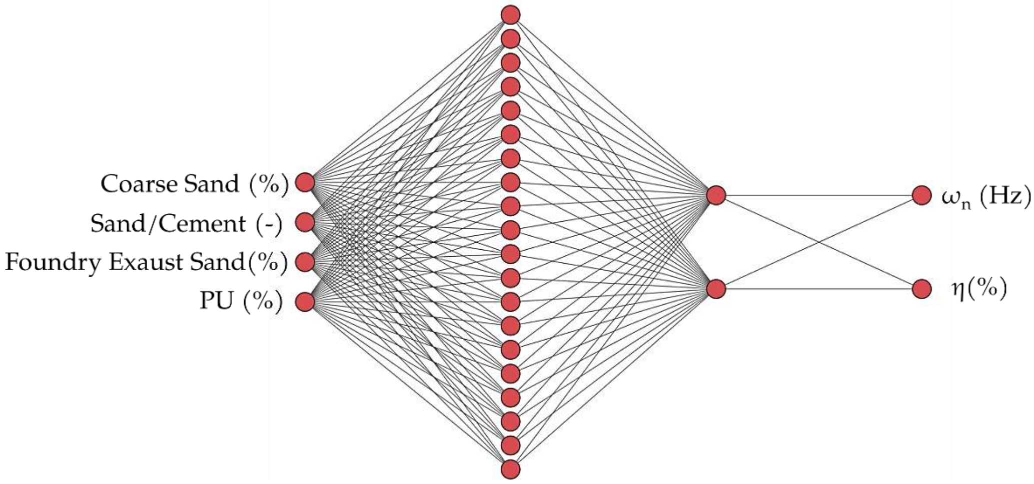

Figure 5-14: The architecture used for the ANN.

The multilayer feedforward network with backpropagation training technique and supervised learning was adopted to obtain high performance. The network is trained using this approach, and the faults it generates are passed down to the previous layers until they are inconsequential (Haykin, 2009). On the ANN architecture, the Levenberg-Marquardt algorithm was utilized to promote a rapid convergence rate and proper training. Table 5-5 shows the additional parameters employed in the ANN generated here.

Table 5-5: Optimal ANN configuration considering the modal behavior prediction

Parameters	ANN model for MPUFES
Learning algorithm	Levenberg-Marquardt
Activation function (hidden layers)	Hyperbolic tangent
Activation function (output layers)	Linear
Mean squared error	0.10
Training data	70%
Max number of iterations	2000
Learning rate	0.30

In this study, a linear regression analysis was developed in order to verify the correlation between the data sets by ANN for training and validation. The fit is measured by the coefficient of determination (R^2), which has values between $0 \leq R^2 \leq 1$, close to one, the coefficient demonstrates that the variables accurately describe the regression model [166]. As is shown in Figure 5-15c, the coefficient of determination was close to one (0.99723), indicating that the observed ANN data are reliable and accurate in representing the behavior of the MPUFES modal model. The overall training results are shown in Figure 5-15a, with the best training performance measured by the MSE obtained at the eighth interaction (Epoch 8). This demonstrates that the ANN has a fast convergence that helps reduce training time. In Figure 5-15b, the error histogram describes the difference between the target value and value after training the neural net. The better the neural net is trained, the more concentrated the histogram gets near zero.

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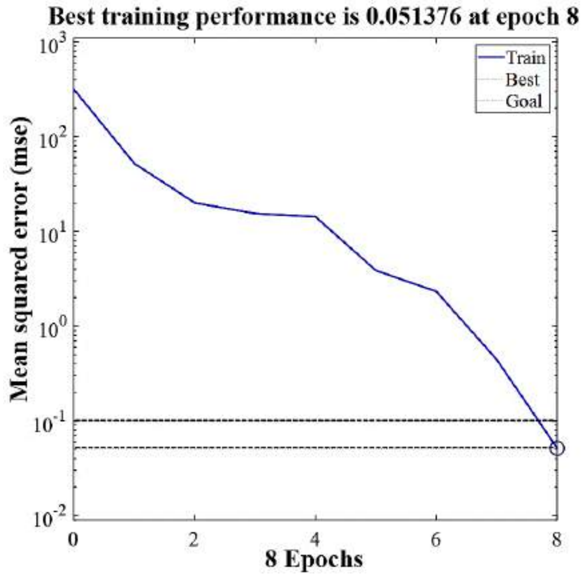
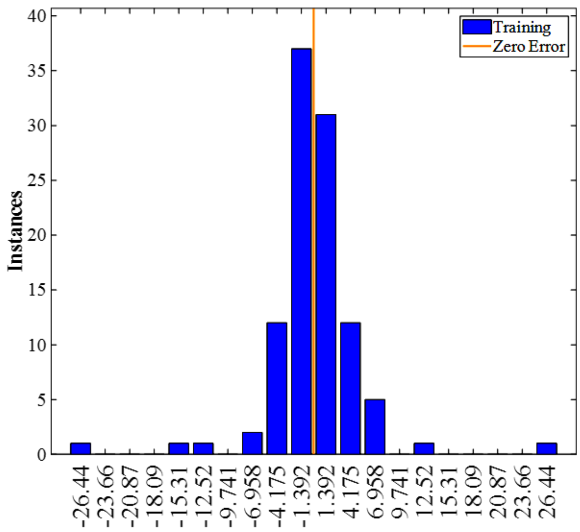
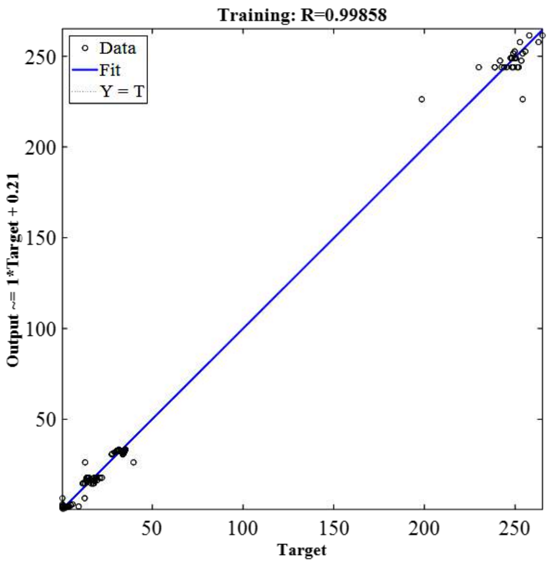


(a)



(b)



(c)

Figure 5-15: Results of ANN global training considering: (a) the best training performance, (b) the histogram of error values, and (c) the linear regression analysis with coefficient of determination.

Finally, Figure 5-16 shows the prediction results of the ANN compared to the test data (not known by the ANN model) in graphical form. Test data is information that has not been used in network training. This test is the most effective approach to determine whether or not the network has been properly taught because they are unknown. The results showed that the net performed as expected. It is clear that, particularly in the circumstances of damping and natural frequency, the net response follows the same pattern as the real response. The net displayed low errors even for the extreme values of the variables S/C, SC, FES, and PU.

Plots are performed by pairs of decision variables for graphical visualization. From the results obtained, complementary to Figure 5-14, it is possible to infer about some aspects: i) The natural frequency has low variation as a function of the sand/cement and coarse sand rate and is more sensitive to the presence of PU (Figure 5-16c); ii) the second modal response, damping, presents a linear behavior as a function of the sand/cement and coarse sand ratio. A sand/cement ratio ~3 with high coarse sand ratios contribute to an increase in structural damping. Considering the parameters of PU and foundry sand, the same pattern is observed for the damping as observed for the natural frequency. The PU variable introduced nonlinearities in the system.

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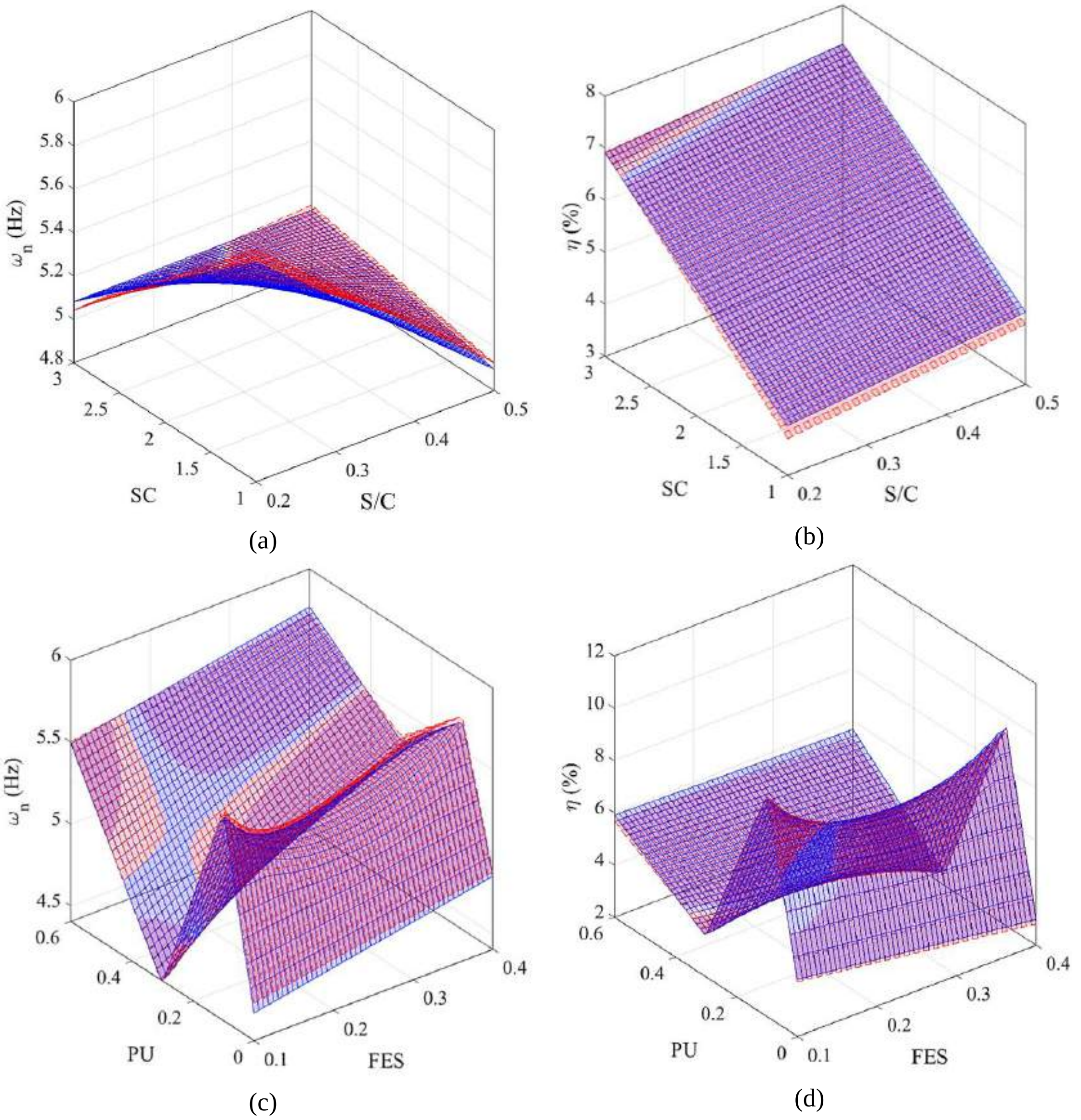

Figure 5-16: ANN graphical results of (a) Natural Frequency (ω_n) by SC and S/C, (b) damping (η) by SC and S/C, (c) Natural Frequency (ω_n) by PU and FES (d) damping (η) by PU and FES. (Legend: — Real, — ANN predicted)

The authors reached significant results using analysis parameters to investigate the response to deformation in engineering structures, an approach similar to those adopted in this study [238–240]. Once the linear regression models and ANN were compared to evaluate the accuracy of the parameters.

5.6 Conclusions

The modal response of mortar mixtures with polyurethane incorporation and used foundation sand was examined experimentally to determine the effects of design factors. During the free vibration testing, which isolated the effect of PU, the natural frequency of MPUFES changed from increasing by 11.6% to decreasing by -21.7%. While under the influence of FES alone, the natural frequency varied with an increase of 1.8% and a drop of -4.0%.

The damping factor testing revealed a pattern resembling the natural frequency with more fluctuation. If you focus on the impact of PU alone, you can see variety with increases of 78.7% and decreases of -47.3%. When the impact of FES is taken out, variation with increases of 26.0% and decreases of -2.2% can be seen.

Additionally, the experimental data when compared to the statistical findings revealed that the MPUFES design variables significantly influenced the rise and fall of the modal characteristics, natural frequency, and damping factor of the waste-filled mortars in comparison to the waste-free mortars. Furthermore, the ANN demonstrated outstanding modal response prediction performance with the experimental tests run in this inquiry. This ANN was trained using a subset of the experimental data set.

To better understand the impacts and behavior of these wastes on the modal response of reinforced mortars, additional analysis and testing are needed. Additional research on free and forced vibration using this method, the design characteristics of those tested, and the production of the mortars utilized and selected in this study may aid in elucidating the mechanisms leading to the results attained.

As a result, it was shown that, the modal or dynamic analysis of mortar with polyurethane waste and foundry sand from exhaust can be predicted using an ANN model with a not too large number of samples (108 samples) with small errors. This study contributes to experimental work in the form of mapping mixture proportions and by the variations of modal parameters such as natural frequency and loss factor. Numerically, this can be achieved without wasting material, thus reducing design costs. In conclusion, RNNs are robust and a viable tool with potential to predict cementitious mortar properties.

GENERAL CONCLUSION

This study consists of the development, optimization and testing of the incorporation of industrial waste such as polyurethane powder and foundry exhaust sand from exhaust into portland cement composites such as mortar. To fulfill the proposed objectives, several steps were followed and presented throughout the study.

The field of study related to composite materials of portland cement and industrial waste, more specifically polymeric waste, has shown great growth in recent years. This behavior can be attributed to the great awareness of the environment and also to the technological advance of cementitious materials, such as mortars and concrete for different types of use in civil construction.

The incorporation of powdered polyurethane residue proved to be sustainable within a restricted range of substitution. The recommended replacement of PU by natural sand is by volume, due to the small specific mass of PU compared to natural sand. As concluded throughout the studies, PU can significantly modify the mechanical, physical and dynamic characteristics of the mortar tested.

The incorporation of foundry exhaust sand (FES) proved to be adequate within a wide substitution range. Unlike PU, the FES residue can be used in higher percentages (25%), affecting more smoothly the measured properties.

In this way, all the variables used were effective, each one with its weight and within a certain spectrum of incorporation. The mathematical models obtained can predict a wide range of properties required for different scenarios.

Important answers were obtained from the statistical analysis. First is the confirmation of the mathematical model through optimization experiments with adequate adjustment. Second is that through different optimization parameters, the effect of all parameters combined as well as the isolated effect of parameters can be characterized by statistical analysis. Finally, the response surface obtained illustrates how the factors are influenced by the input parameters. The response surface shows that the behavior can be linear or non-linear and will depend on the chosen embedding percentage.

As a suggestion for future work:

- Investigation for the applicability of other polymeric residues within the model obtained, such as Polyethylene (PE), Polypropylene (PP), Polystyrene (PS), Polyamides (PA), Polyvinyl Chloride (PVC), Polycarbonate (PC) among other types of residues produced on a large scale.
- Evaluate the durability of mortars and concretes with residues in unsanitary scenarios, such as an environment rich in chlorides, sulfates and passive agents that chemical reactions capable of damaging the chemical structure of the resulting composite.
- Evaluate the applicability of composites produced in real construction situations, such as masonry coating, block laying, floor regularization and activities related to civil construction.

PUBLICATIONS

The publications resulting out of this thesis are listed below:

L.R. Roque-Silva, P.M. Alves, M.H.B. Souza, G.Z. Costal, R.M. Martins, P.C. Gonçalves, P. Capellato, M.G.A. Ranieri, R.G. Torres, M.L.N.M. Melo, V.C. dos Santos. Analysis of the Scientific Production of Cementitious Composites with Recycled Polymeric Materials. Book: **Recycling of Plastics, Metals, and Their Composites**. 2021-11-29. Book chapter. DOI: 10.1201/9781003148760-20. Part of ISBN: 9781003148760

Lucas Ramon Roque da Silva, Matheus Brendon Francisco, Valquíria Claret dos Santos, Paulo Cesar Gonçalves, Rosa Cristina Cecche Lintz, Guilherme Ferreira Gomes, Mirian de Lourdes Noronha Motta. **Design of experiments to optimize use of polyurethane residue in cement composite**. Journal of Sustainable Cement-Based Materials - Taylor & Francis Online. ISSN 2165-0381 (In submission process).

Lucas Ramon Roque da Silva, Matheus Brendon Francisco, Roberta Moraes Martins, Paulo Cesar Gonçalves, Valquíria Claret dos Santos, Guilherme Ferreira Gomes, Mirian de Lourdes Noronha Motta Melo, RSM-based modeling and optimization of cementitious composites with polyurethane powder waste and foundry exhaust sand. **Journal of Materials in Civil Engineering - Journals – ASCE**. ISSN (print): 0899-1561. ISSN (online): 1943-5533 (Accepted).

Lucas Ramon Roque da Silva, Paulo Cesar Gonçalves, Valquíria Claret dos Santos, Mirian de Lourdes Noronha Motta Melo, Guilherme Ferreira Gomes. An experimental dynamic study of cement mortar with polyurethane residues and foundry sand. **Engineering Structures - Journals – Elsevier.** ISSN: 0141-0296 (under peer review).

PUBLICATIONS AS CO-AUTHOR

M.A.B.Martins, L.R.R.Silva, B.H.B.Kuffner, R.M.Barros, M.L.N.M.Melo. Behavior of high strength self-compacting concrete with marble/granite processing waste and waste foundry exhaust sand, subjected to chemical attacks. **Construction and Building Materials**. Volume 323, 14 March 2022, 126492. https://doi.org/10.1016/j.conbuildmat.2022.126492.

M.A.Martins, R.M.Barros, L.R.R.Silva, V.C.Santos, R.C.C.Lintz, L.A.Gachet, M.L.Melo, C.B.Martinez. Durability indicators of high-strength self-compacting concrete with marble and granite wastes and waste foundry exhaust sand using electrochemical tests. **Construction and Building Materials.** Volume 317, 24 January 2022, 125907. https://doi.org/10.1016/j.conbuildmat.2021.125907.

M.G.A. Ranieri, P. Capellato, M.A. de B. Martins, V.C. dos Santos, P.C. Gonçalves, L.R. Roque-Silva, M.L.M. Melo, A. da S. Mello. Cementitious Composites for Civil Construction Made with Marble and Granite Waste. **Book: Recycling of Plastics, Metals, and Their Composites**. 2021/11/21. Book chapter DOI: 10.1201/9781003148760-21. Part of ISBN: 9781003148760.

M.A.B.Martins, F.B.Pinto, D.Werdine, L.R.R.Silva, C.V. Santos, P.C. Gonçalves, M.L.M. Melo, R.M. Barros. **Procedures for Additions of Wastes to Cementitious Composites – A Review**. Recycling of Plastics, Metals, and Their Composites. 2021-11-21. Book chapter DOI: 10.1201/9781003148760-19. Part of ISBN: 9781003148760.

Matheus Brendon Francisco, João Luiz Junho Pereira, Guilherme Antônio Oliver, Lucas Ramon Roque da Silva, Sebastião Simões Cunha Jr, Guilherme Ferreira Gomes. **A review on the energy absorption response and structural applications of auxetic structures.** Mechanics of Advanced Materials and Structures. https://doi.org/10.1080/15376494.2021.1966143.

Maria Auxiliadora de Barros Martins, Lucas Ramon Roque da Silva, Maria Gabriela A. Ranieri, Regina Mambeli Barros, Valquíria Claret dos Santos, Paulo César Gonçalves, Márcia Regina Baldissera Rodrigues, Rosa Cristina Cecche Lintz, Luísa Andréa Gachet, Carlos Barreira Martinez, Mirian de Lourdes Noronha Motta Melo. **Physical and Chemical Properties of Waste Foundry Exhaust Sand for Use in Self-Compacting Concrete**. Materials 2021, 14(19), 5629; https://doi.org/10.3390/ma14195629.

Francisco, M., Roque, L., Pereira, J., Machado, S., da Cunha Jr, S.S.,Gomes, G.F. **A statistical analysis of high-performance prosthetic isogrid composite tubes using response surface method.** Engineering Computations, 2021, Vol. 38 No. 6, pp. 2481-2504. https://doi.org/10.1108/EC-04-2020-0222

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